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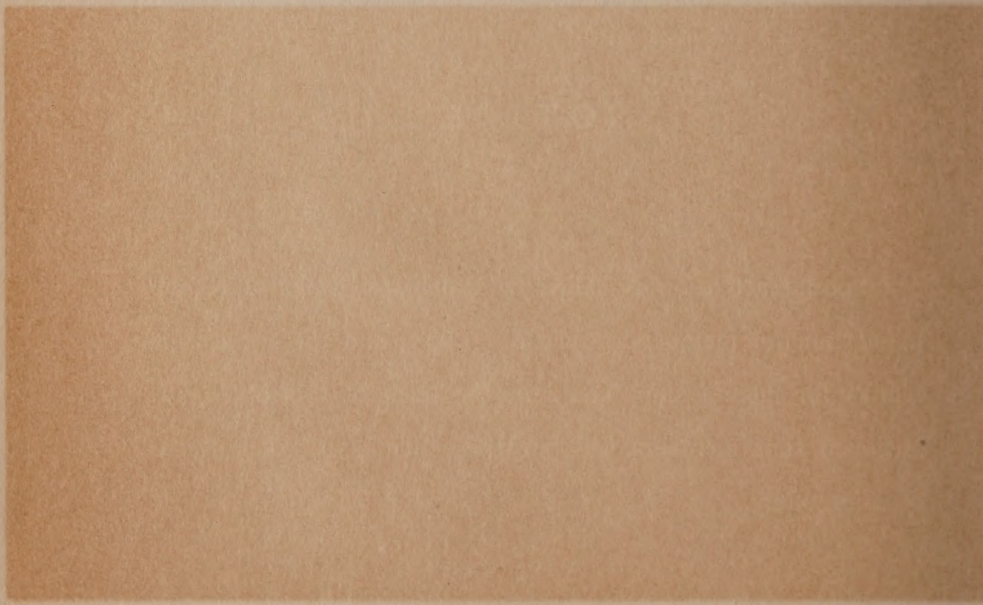

NOTICE

Special attention is called to the blank letter of recommendation for membership in the Society, and the application blanks for membership bound in the back of this book. It is hoped that members will put these blanks to good use as occasion offers.

AMERICAN SOCIETY

FOR

TESTING MATERIALS

ORGANIZED IN 1898
INCORPORATED IN 1902

A.S.T.M. STANDARDS

(ISSUED TRIENNIALLY)

1927

PART II. NON-METALLIC MATERIALS

PUBLISHED BY
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FOREWORD

The 1927 Book of Standards is published in two parts, Part I covering Metals and Part II covering Non-Metallic Materials. In these two Parts appear the 340 standard specifications, methods of test, definitions of terms and recommended practices in effect at the time of publication. A complete list of these standards is given on page 943.

This volume comprises Part II and contains 196 standards, of which 193 are standards relating specifically to non-metallic materials and 3 are standards of a general nature applying to both metals and non-metals, which latter standards for convenience are included in both Parts.

The Book of Standards is issued triennially. Those standards which are adopted by the Society in the intervening years are published in supplements to the Book of Standards. In accordance with this policy there will be issued a Supplementary Pamphlet of A.S.T.M. Standards Adopted in 1928 and a second Supplementary Pamphlet of A.S.T.M. Standards Adopted in 1929, which will contain the standards approved by the Society in those years. The next edition of the Book of Standards will be published in 1930.

Before a standard receives the formal approval of the Society it is published for one or more years as a tentative standard. The tentative standards submitted or revised in any given year appear in the Proceedings of the Society for that year. In addition all tentative standards in effect in any given year are published collectively in a Book of A.S.T.M. Tentative Standards which is issued each year about October 15. Criticisms of such tentative standards are solicited, of which the standing committee responsible for the preparation and revision of the standards in question takes due cognizance before recommending final adoption as standard by the Society. The procedure for the submission and adoption of standards is given in the Regulations Governing Standing Committees appearing on pages 935 to 942, inclusive. A list of the current tentative standards is printed in this volume on pages 958 to 967. Corresponding lists of tentative standards effective in subsequent years will appear in the supplements to the Book of Standards for those years.

GUIDE TO THE USE

OF THE

BOOK OF STANDARDS

The standards in this Book are assembled in a sequence determined by the specific materials or products to which they apply. For convenience in using the Book of Standards each Part is provided with **two Tables of Contents,** one a listing of the standards in the order in which they appear in the volume grouped according to the subjects to which they relate, the other a listing of the standards **in numeric sequence** of their serial designations.

In addition to the Tables of Contents a complete **Subject Index** appears in the back of each Part. (For the Index to this Part, see page 968.) Each standard has been indexed under the principal subjects covered by the standard. The principal words in each item of the index have been printed in bold face type to facilitate finding the item desired. This index should be the most convenient means of locating any standard appearing in the volume, especially if only the general subject matter covered is known.

As mentioned in the Foreword, a complete **list of all standards and tentative standards** of the Society in effect as of September 1, 1927, is included in this volume, pages 943 to 967. Corresponding lists of standards and tentative standards in effect as of September 1, 1928, and September 1, 1929, respectively, will appear in the supplements to the Book of Standards for those years.

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The standards are grouped according to the materials to which they apply. Duplicate listings appear in italics.

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STANDARD SPECIFICATIONS AND TESTS FOR PORTLAND CEMENT[1]

Serial Designation: C 9 – 26

These specifications are issued under the fixed designation C 9; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1904; REVISED, 1908, 1909, 1916, 1920, 1926.

SPECIFICATIONS

Definition.

1. Portland cement is the product obtained by finely pulverizing clinker produced by calcining to incipient fusion an intimate and properly proportioned mixture of argillaceous and calcareous materials, with no additions subsequent to calcination excepting water and calcined or uncalcined gypsum.

CHEMICAL PROPERTIES

Chemical Limits.

2. The following limits shall not be exceeded:

Loss on ignition, per cent	4.00
Insoluble residue, per cent	0.85
Sulfuric anhydride (SO_3), per cent	2.00
Magnesia (MgO), per cent	5.00

PHYSICAL PROPERTIES

Fineness.

3. The residue on a standard No. 200 sieve shall not exceed 22 per cent by weight.

Soundness.

4. A pat of neat cement shall remain firm and hard, and show no signs of distortion, cracking, checking, or disintegration in the steam test for soundness.

Time of Setting.

5. The cement shall not develop initial set in less than 45 minutes when the Vicat needle is used or 60 minutes when the Gillmore needle is used. Final set shall be attained within 10 hours.

Tensile Strength.

6. The average tensile strength in pounds per square inch of not less than three standard mortar briquets (see Section 47) composed of one part of cement and three parts of standard sand, by weight, shall be equal to or higher than the following:

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

AGE AT TEST, DAYS	STORAGE OF BRIQUETS	TENSILE STRENGTH, LB. PER SQ. IN.
7	1 day in moist air, 6 days in water	225
28	1 day in moist air, 27 days in water	325

7. The average tensile strength of standard mortar at 28 days shall be higher than the strength at 7 days.

PACKING, MARKING AND STORAGE

Packages and Marking.

8. The cement shall be delivered in packages as specified with the brand and name of the manufacturer plainly marked thereon, unless shipped in bulk. When shipped in bulk, this information shall be contained in the shipping advices accompanying the shipment. A bag shall contain 94 lb. net. A barrel shall contain 376 lb. net. All packages shall be in good condition at the time of inspection.

Storage.

9. The cement shall be stored in such a manner as to permit easy access for proper inspection and identification of each shipment, and in a suitable weather-tight building which will protect the cement from dampness.

INSPECTION

Inspection.

10. Every facility shall be provided the purchaser for careful sampling and inspection at either the mill or at the site of the work, as may be specified by the purchaser. At least 12 days from the time of sampling shall be allowed for the completion of the 7-day test, and at least 33 days shall be allowed for the completion of the 28-day test. The cement shall be tested in accordance with the methods hereinafter prescribed. The 28-day test need not be made if waived by the purchaser.

REJECTION

Rejection.

11. The cement may be rejected if it fails to meet any of the requirements of these specifications.

12. Cement remaining in storage prior to shipment for a period greater than 6 months after test shall be retested and shall be rejected if it fails to meet any of the requirements of these specifications.

13. Cement shall not be rejected on account of failure to meet the fineness requirement if upon retest after drying at 100° C. for one hour it meets this requirement.

14. Cement failing to meet the test for soundness in steam may be accepted if it passes a retest using a new sample at any time within 28 days thereafter. The provisional acceptance of the cement at the mill shall not deprive the purchaser of the right of rejection on a retest

of soundness and time of setting at the time of delivery of cement to the purchaser.

15. Packages varying more than 5 per cent from the specified weight may be rejected; and if the average weight of packages in any shipment, as shown by weighing 50 packages taken at random, is less than that specified, the entire shipment may be rejected.

TESTS

SAMPLING

Number of Samples.

16. Tests may be made on individual or composite samples as may be ordered. Each test sample should weigh at least 4 lb.

17. (*a*) *Individual Sample.*—If sampled in cars, one test sample shall be taken from each 50 bbl. or fraction thereof. If sampled in bins one sample shall represent each 200 bbl. unless otherwise specified by the purchaser.

(*b*) *Composite Sample.*—If sampled in cars, one sample shall be taken from one sack in each 40 sacks (or 1 bbl. in each 10 bbl.) and combined to form one test sample. If sampled in bins or warehouses one test sample shall represent not more than 200 bbl. unless otherwise specified by the purchaser.

Method of Sampling.

18. Cement may be sampled at the mill by any of the following methods that may be practicable, as specified:

(*a*) *From the Conveyor Delivering to the Bin.*—At least 4 lb. of cement shall be taken from approximately each 100 bbl. passing over the conveyor. This may be secured by taking the entire test sample at a single operation, known as the "Grab Method," or by combining several portions taken at regular intervals, known as the "Composite Method."

(*b*) *From Filled Bins by Means of Proper Sampling Tubes.*—Tubes inserted vertically may be used for sampling cement to a maximum depth of 10 ft. Tubes inserted horizontally may be used where the construction of the bin permits. Samples shall be taken from points well distributed over the face of the bin.

(*c*) *From Filled Bins at Points of Discharge.*—Sufficient cement shall be drawn from the discharge openings to obtain samples representative of the cement contained in the bin, as determined by the appearance at the discharge openings of indicators placed on the surface of the cement directly above these openings before drawing of the cement is started.

19. The sampling shall be done by or under the direction of a responsible representative of the purchaser.

Treatment of Sample. 20. Samples preferably shall be shipped and stored in moisture-proof, air-tight containers. Samples shall be passed through a sieve having 20 meshes per linear inch in order to thoroughly mix the sample, break up lumps and remove foreign materials.

CHEMICAL ANALYSIS

Loss on Ignition

Method. 21. One gram of cement shall be heated in a weighed covered platinum crucible, of 20 to 25-cc. capacity, as follows, using either Method (*a*) or (*b*) as specified:

Method (a):

The crucible shall be placed in a hole in an asbestos board, clamped horizontally so that about three-fifths of the crucible projects below, and blasted at a full red heat for 15 minutes with an inclined flame; the loss in weight shall be checked by a second blasting for 5 minutes. Care shall be taken to wipe off particles of asbestos that may adhere to the crucible when withdrawn from the hole in the board. Greater neatness and shortening of the time of heating are secured by making a hole to fit the crucible in a circular disk of sheet platinum and placing this disk over a somewhat larger hole in an asbestos board.

Method (b):

The crucible shall be placed in a muffle at any temperature between 900 and 1000° C. for 15 minutes and the loss in weight shall be checked by a second heating for 5 minutes.

Permissible Variation. 22. A permissible variation of 0.25 will be allowed, and all results in excess of the specified limit but within this permissible variation shall be reported as 4 per cent.

Insoluble Residue

Method. 23. To a 1-g. sample of cement shall be added 25 cc. of water and 5 cc. of concentrated hydrochloric acid (sp. gr. 1.19). Material shall be ground with the flattened end of a glass rod until it is evident that the decomposition of the cement is complete. The solution shall then be diluted to 50 cc. and digested on a steam bath for 15 minutes. The residue shall be filtered, washed with cold water and the filter paper and contents digested in about 30 cc. of a 5-per-cent solution of sodium carbonate, the liquid being held at a temperature just short of boil-

ing for 15 minutes. The remaining residue shall be filtered, washed with hot water, then with a few drops of hot hydrochloric acid (1:9) and finally with hot water, then ignited at a red heat and weighed as the insoluble residue.

Permissible Variation.

24. A permissible variation of 0.15 will be allowed, and all results in excess of the specified limit but within this permissible variation shall be reported as 0.85 per cent.

SULFURIC ANHYDRIDE

Method.

25. To a 1-g. sample of cement shall be added 25 cc. of water and 5 cc. of concentrated hydrochloric acid (sp. gr. 1.19). Material shall be ground with the flattened end of a glass rod until it is evident that decomposition of the cement is complete. The solution shall be diluted to 50 cc. and digested on a steam bath for 15 minutes, filtered, and the residue washed thoroughly with hot water. The solution shall be diluted to 250 cc., heated to boiling, and 10 cc. of a hot 10-per-cent solution of barium chloride shall be added slowly drop by drop, from a pipette and the boiling continued until the precipitate is well formed. The solution shall then be digested on the steam bath at least three hours, preferably over night. The precipitate shall be filtered, washed and the paper and contents placed in a weighed platinum crucible and the paper slowly charred and consumed without flaming. The barium sulfate shall then be ignited and weighed. The weight obtained multiplied by 34.3 gives the percentage of sulfuric anhydride. The acid filtrate obtained in the determination of the insoluble residue may be used for the estimation of sulfuric anhydride instead of using a separate sample.

Permissible Variation.

26. A permissible variation of 0.10 will be allowed, and all results in excess of the specified limit but within this permissible variation shall be reported as 2.00 per cent.

MAGNESIA

Method.

27. To 0.5 g. of the cement in an evaporating dish shall be added 10 cc. of water to prevent lumping and then 10 cc. of concentrated hydrochloric acid (sp. gr. 1.19). The material shall be ground with the flattened end of a glass rod until attack is complete. The solution shall then be evaporated to complete dryness on a steam or water bath. To hasten dehydration, the residue may be heated to 150° C. or even 200° C. for one-half to one hour. The residue shall be treated with 10 cc. of hydrochloric acid diluted with an equal amount of water. The dish shall be covered, and the solution digested for 10 minutes on a steam

bath or water bath. The solution shall be diluted to 75 cc., filtered into a beaker, and the separated silica washed thoroughly with hot water (Note 1). Five cubic centimeters of concentrated hydrochloric acid and two drops of methyl red indicator (0.2-per-cent alcoholic solution) shall be added to the filtrate (about 250 cc.) (Note 2).

NOTES

1. Since this procedure does not involve the determination of silica, a second evaporation is unnecessary.

2. Manganese, if present, is distributed between the precipitates of iron and alumina, calcium and magnesium. In the amounts usually present in portland cement, it may be neglected. If it is desired to determine the small amount of manganese that may be present in the magnesium pyrophosphate, this may be done colorimetrically as described in the U. S. Geological Survey *Bulletin No. 700*, p. 153. If present in larger amounts, manganese should be precipitated with the iron and alumina, preferably by the persulfate method (U. S. Geological Survey *Bulletin No. 700*, p. 112). If this method is used more aluminum passes into solution than in the method above described.

Dilute ammonium hydroxide shall be added dropwise until the color of the solution changes to a distinct yellow. The solution shall be boiled for one or two minutes and filtered at once. The beaker and precipitate shall be washed slightly with a hot 2-per-cent solution of ammonium chloride (or ammonium nitrate). Setting aside the filtrate, the precipitate shall be transferred by a jet of hot water to the precipitating vessel and dissolved in 10 cc. of hot hydrochloric acid. The paper shall then be extracted with acid, the solution and washings being added to the solution of the precipitate. The aluminum and iron shall then be reprecipitated at boiling heat by ammonium hydroxide as before in a volume of about 100 cc. and the second precipitate shall be collected and washed with a hot 2-per-cent solution of ammonium chloride (or ammonium nitrate) on the filter used in the first instance, if this is still intact. To the combined filtrates from the hydroxides of iron and aluminum, reduced in volume if need be, 1 cc. of ammonium hydroxide shall be added, the solution brought to boiling, 25 cc. of a saturated solution of boiling ammonium oxalate added, and the boiling continued until the precipitated calcium oxalate has assumed a well defined granular form. The precipitate after 1 hour shall be filtered and washed, and the filtrate set aside. The filter shall be placed wet in a platinum crucible, and the paper burned off over a small flame of a Bunsen burner; after ignition, it shall be cautiously moistened with water, redissolved in hydrochloric acid and the solution diluted to 100 cc.; ammonia shall be added in slight excess, the liquid boiled, and filtered if a precipitate appears. The lime shall then be reprecipitated by ammonium oxalate, allowed to stand till settled,

filtered and washed. The combined filtrates from the calcium precipitates shall be acidified with hydrochloric acid, concentrated on the steam bath to about 150 cc. and made slightly alkaline with ammonium hydroxide, boiled and filtered (to remove a little iron and aluminum, and perhaps calcium). When cool, the solution shall be acidified with hydrochloric acid, 10 cc. of saturated solution of sodium-ammonium-hydrogen phosphate added, and ammonia drop by drop, with constant stirring. When the crystallin ammonium-magnesium orthophosphate has formed, 5 cc. excess ammonia shall be added. The solution shall be set aside for not less than 4 hours, preferably over night, in a cool place, filtered and washed with water containing 2.5 per cent NH_3. The precipitate shall be dissolved in a small quantity of hot hydrochloric acid, the solution diluted to about 100 cc., 1 cc. of a saturated solution of sodium-ammonium-hydrogen phosphate added, and ammonia drop by drop, with constant stirring, until the precipitate is again formed as described and the ammonia is in moderate excess. The precipitate shall then be allowed to stand about 2 hours, filtered and washed as before. The paper and contents shall be placed in a weighed platinum crucible, the paper slowly charred, and the resulting carbon carefully burned off. The precipitate shall then be ignited to constant weight over a Meker burner, or a blast not strong enough to soften or melt the pyrophosphate. The weight of magnesium pyrophosphate obtained multiplied by 72.5 gives the percentage of magnesia. The precipitate so obtained always contains some calcium and usually small quantities of iron, aluminum, and manganese as phosphates.

28. A permissible variation of 0.4 will be allowed, and all results in excess of the specified limit but within this permissible variation shall be reported as 5.00 per cent. **Permissible Variation.**

DETERMINATION OF FINENESS

29. Wire cloth for standard sieves for cement shall be woven (not twilled) from brass, bronze, or other suitable wire, and mounted without distortion on frames about 2 in. below the top of the frame. The joint between the cloth and frame shall be smoothly filled with solder to prevent lodging of the cement. The sieve frames shall be circular, approximately 8 in. in diameter, and may be provided with a pan and cover. **Apparatus.**

30. A standard No. 200 sieve is one having nominally a 0.0029-in. opening, certified by the U. S. Bureau of Standards, and conforming to the specifications for this sieve in the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the

American Society for Testing Materials.[1] The correction to the sieving value of the sieve shall be determined by sieving tests made in conformity with the standard specifications for these tests on a standardized cement which gives a residue of about 20 per cent on the No. 200 sieve.

Method. 31. The test shall be made with 50 g. of cement. The sieve shall be thoroughly clean and dry. The cement shall be placed on the No. 200 sieve, with pan and cover attached, if desired, and shall be held in one hand in a slightly inclined position so that the sample will be well distributed over the sieve, at the same time gently striking the side about 150 times per minute against the palm of the other hand on the up stroke. The sieve shall be turned every 25 strokes about one-sixth of a revolution in the same direction. The operation shall continue until not more than 0.05 g. passes through in one minute of continuous sieving (Note 1). The fineness shall be determined from the weight of the residue on the sieve expressed as a percentage of the weight of the original sample, applying the sieve correction (Note 2).

NOTES

1. The essential points in the sieving operation may be summarized as follows:
   (1) Rotation of the sieve throughout the process.
   (2) Guarding against loss of material. Sieve over white paper and always tap the sieve gently.
   (3) Use of a balance which will give results correct within 5 mg. and sufficiently sensitive so that the rest point will be deflected at least two divisions of the scale for an added load of 5 mg.
   (4) Washers, shot and slugs should never be used on the sieve.
   (5) Excessive humidity interferes with good sieving. It tends to decrease the percentage of cement passing the sieve, and in general, to produce irregular results.

2. A plus (+) sieve correction indicates the amount to be added to and a minus (−) sieve correction the amount to be subtracted from the per cent passing the sieve to obtain the true fineness. The per cent passing is the amount obtained by subtracting the per cent residue from 100. The corrections are used in the reverse order when applied to the residues direct.

32. Mechanical sieving devices may be used, but the cement shall not be rejected if it meets the fineness requirement when tested by the hand method described in Section 31.

MIXING CEMENT PASTES AND MORTARS

Method. 33. The quantities of dry materials to be mixed at one time shall be 500 g. for neat cement mixtures and 1000 g. for mortar mixtures. The proportions of cement or cement and sand shall be stated by

[1] See p. 917.

weight in grams of the dry materials; the quantity of water shall be expressed in cubic centimeters (1 cc. of water = 1 g.). The dry materials shall be weighed, placed upon a non-absorbent surface, thoroughly mixed dry if sand is used, and a crater formed in the center, into which the proper percentage of clean water shall be poured; the

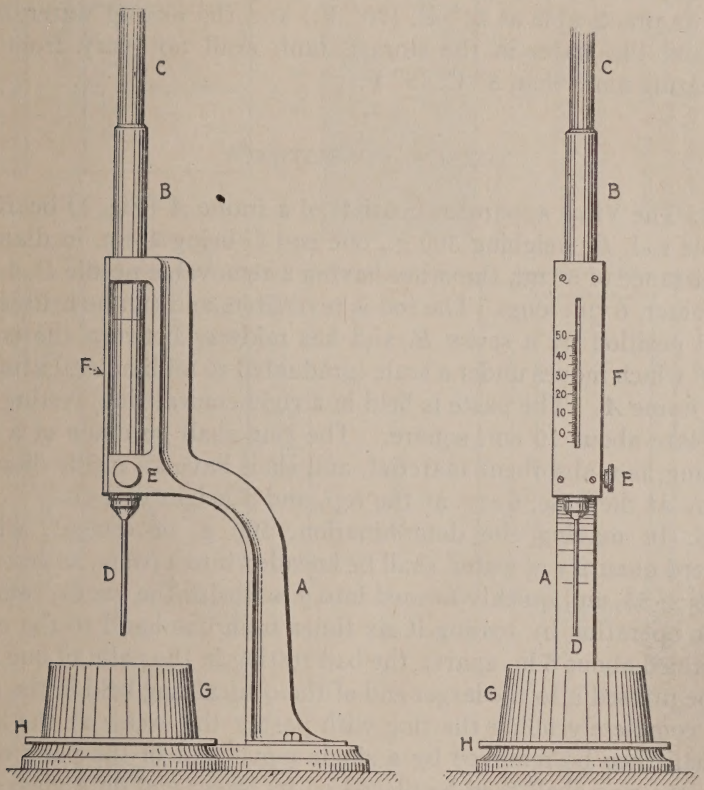


FIG. 1.—Vicat Apparatus.

material on the outer edge shall be turned into the crater by the aid of a trowel. After an interval of ½ minute for the absorption of the water the operation shall be completed by continuous, vigorous mixing, squeezing and kneading with the hands for at least one minute. During the operation of mixing, the hands shall be protected by rubber gloves.

NOTE.—In order to secure uniformity in the results of tests for the time of setting and tensile strength the manner of mixing above described shall be carefully

followed. At least one minute is necessary to obtain the desired plasticity which is not appreciably affected by continuing the mixing for several minutes. The exact time necessary is dependent upon the personal equation of the operator. The error in mixing shall be on the side of over mixing.

Temperature.

34. The temperature of the room, the materials, the mixing water, the moist closet and storage-tank water shall be maintained as nearly as practicable at 21° C. (70° F.) and the mixing water, moist closet and the water in the storage tank shall not vary from this temperature more than 3° C. (5° F.).

NORMAL CONSISTENCY

Apparatus.

35. The Vicat apparatus consists of a frame *A* (Fig. 1) bearing a movable rod, *B*, weighing 300 g., one end *C* being 1 cm. in diameter for a distance of 6 cm., the other having a removable needle *D*, 1 mm. in diameter, 6 cm. long. The rod is reversible, and can be held in any desired position by a screw *E*, and has midway between the ends a mark *F* which moves under a scale (graduated to millimeters) attached to the frame *A*. The paste is held in a rigid conical ring, resting on a glass plate about 10 cm. square. The ring shall be made of a non-corroding, non-absorbent material, and shall have an inside diameter of 7 cm. at the base, 6 cm. at the top, and a height of 4 cm.

Method.

36. In making the determination, 500 g. of cement, with a measured quantity of water, shall be kneaded into a paste, as described in Section 33, and quickly formed into a ball with the hands, completing the operation by tossing it six times from one hand to the other, maintained about 6 in. apart; the ball resting in the palm of one hand shall be pressed into the larger end of the conical ring held in the other hand, completely filling the ring with paste; the excess at the larger end shall then be removed by a single movement of the palm of the hand; the ring shall then be placed on its larger end on a glass plate and the excess paste at the smaller end sliced off at the top of the ring by a single oblique stroke of a trowel held at a slight angle with the top of the ring, and the top smoothed, if necessary, with a few light touches of the pointed end of the trowel. During these operations care shall be taken not to compress the paste. The paste confined in the ring, resting on the plate, shall be placed under the rod, the larger end of which shall be brought in contact with the surface of the paste; the scale shall then be read, and the rod quickly released. The paste shall be of normal consistency when the rod settles to a point 10 mm. below the original surface in $\frac{1}{2}$ minute after being released. The apparatus shall be free from all vibrations during the test. Trial

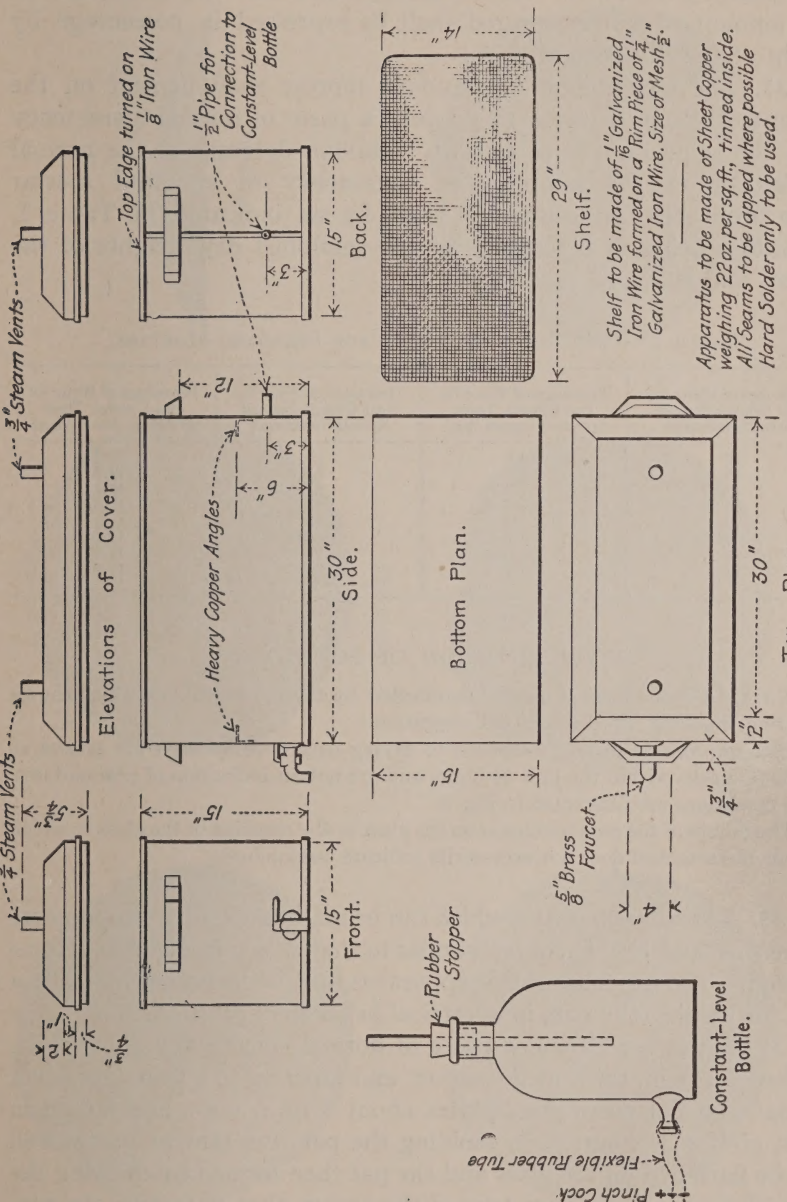


FIG. 2.—Apparatus for Making Soundness Test of Cement.

pastes shall be made with varying percentages of water until the normal consistency is obtained. Each trial shall be made with fresh cement. The amount of water required shall be expressed in percentage by weight of the dry cement.

37. The consistency of standard mortar shall depend on the amount of water required to produce a paste of normal consistency from the same sample of cement. Having determined the normal consistency of the sample, the consistency of standard mortar made from the same sample shall be as indicated in Table I, the values being in percentage of the combined dry weights of the cement and standard sand.

TABLE I.—PERCENTAGE OF WATER FOR STANDARD MORTARS.

Percentage of Water for Neat Cement Paste of Normal Consistency	Percentage of Water for One Cement, Three Standard Ottawa Sand	Percentage of Water for Neat Cement Paste of Normal Consistency	Percentage of Water for One Cement, Three Standard Ottawa Sand
15	9.0	23	10.3
16	9.2	24	10.5
17	9.3	25	10.7
18	9.5	26	10.8
19	9.7	27	11.0
20	9.8	28	11.2
21	10.0	29	11.3
22	10.2	30	11.5

DETERMINATION OF SOUNDNESS

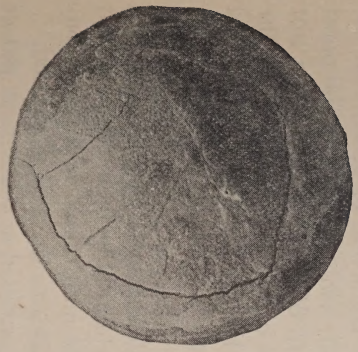
NOTE.—Unsoundness is usually manifested by change in volume which causes distortion, cracking, checking, or disintegration.

Pats improperly made or exposed to drying may develop what are known as shrinkage cracks within the first 24 hours and are not an indication of unsoundness. These conditions are illustrated in Fig. 3.

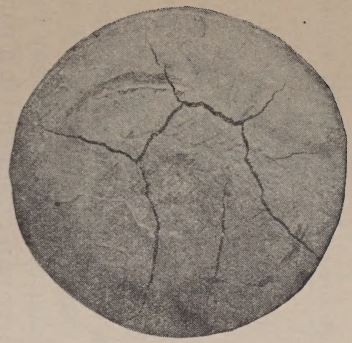
The failure of the pats to remain on the glass or the cracking of the glass to which the pats are attached does not necessarily indicate unsoundness.

Apparatus. 38. A steam apparatus, which can be maintained at a temperature between 98 and 100° C., or one similar to that shown in Fig. 2, is recommended. The capacity of this apparatus may be increased by using a rack for holding the pats in a vertical or inclined position.

Method. 39. A pat from cement paste of normal consistency about 3 in. in diameter, $\frac{1}{2}$ in. thick at the center, and tapering to a thin edge, shall be made on flat clean glass plates about 4 in. square, and stored in moist air for 24 hours. In molding the pat, the cement paste shall first be flattened on the glass and the pat then formed by drawing the trowel from the outer edge toward the center, then flattening the top. The pats used for the time of setting tests by the Gillmore method may be used for soundness tests.

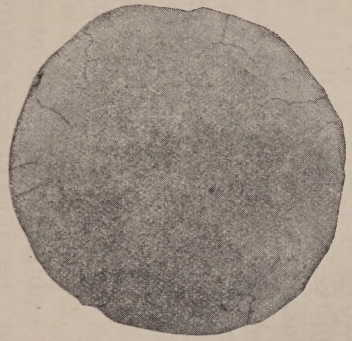


Shrinkage

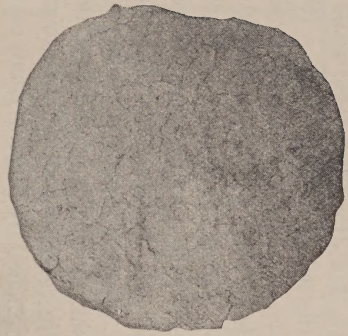


Shrinkage

Distortion



Cracking



Checking

Disintegration

FIG. 3.—Typical Failures in Soundness Test.

40. The pat shall then be placed in an atmosphere of steam at a temperature between 98 and 100° C., upon a suitable support 1 in. above boiling water for 5 hours.

NOTE.—It is important that the specimens be 24 hours old when placed in steam, since variations in their age will produce differences in the results of the steam tests. Particularly noticeable are the effects of steaming pats too soon, for many specimens steamed when only 10 or 12 hours old give apparently satisfactory results, while failure would be observed if they were not placed in steam until 24 hours old.

41. Should the pat leave the plate, distortion may be detected best with a straight edge applied to the surface which was in contact with the plate.

DETERMINATION OF TIME OF SETTING

42. The following are alternate methods, either of which may be used as specified:

Vicat Apparatus.

43. The time of setting shall be determined with the Vicat apparatus described in Section 35. (See Fig. 1.)

Vicat Method.

44. A paste of normal consistency shall be molded in the ring *G* as described in Section 36, and placed under the rod *B*, the smaller end of which shall then be carefully brought in contact with the surface of the paste, and the rod quickly released. The initial set shall be said to have occurred when the needle ceases to pass a point 5 mm. above the glass plate in ½ minute after being released; and the final set, when the needle does not sink visibly into the paste. The test pieces shall be kept in moist air during the test. This may be accomplished by placing them on a rack over water contained in a pan and covered by a damp cloth, kept from contact with them by means of a wire screen; or they may be stored in a moist closet. Care shall be taken to keep the needle clean, as the collection of cement on the sides of the needle retards the penetration, while cement on the point may increase the penetration. The time of setting is affected not only by the percentage and temperature of the water used and the amount of kneading the paste received, but by the temperature and humidity of the air, and its determination is therefore only approximate.

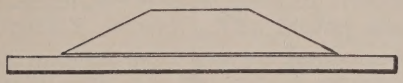
Gillmore Needles.

45. The time of setting shall be determined by the Gillmore needles. The Gillmore needles should preferably be mounted as shown in Fig. 4 (*b*), and the needle ends shall be maintained in a clean condition and shall be plane and at right angles to the axis of the rod.

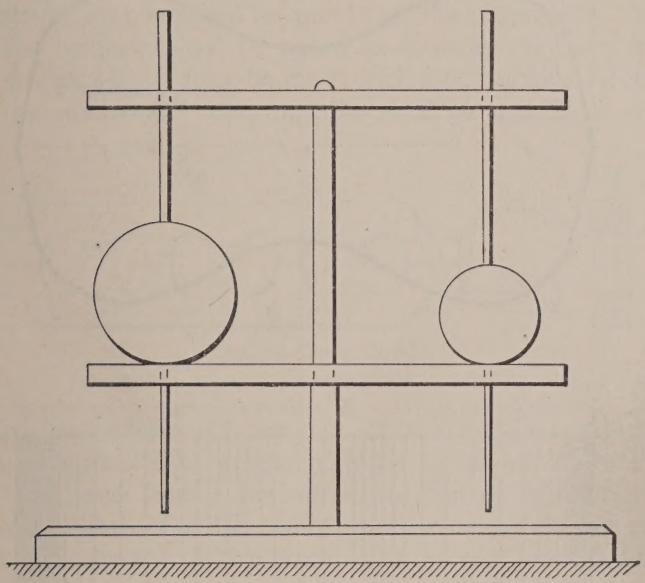
Gillmore Method.

46. The time of setting shall be determined as follows: A pat of neat cement paste about 3 in. in diameter and ½ in. in thickness with a flat top (Fig. 4 (*a*)), mixed to a normal consistency, shall be kept in

moist air at a temperature maintained as nearly as practicable at 21° C. (70° F.). The cement shall be considered to have acquired its initial set when the pat will bear, without appreciable indentation, the Gillmore needle $\frac{1}{12}$ in. in diameter, loaded to weigh $\frac{1}{4}$ lb. The final set has been acquired when the pat will bear, without appreciable indentation, the Gillmore needle $\frac{1}{24}$ in. in diameter, loaded to weigh 1



(*a*) Pat with Top Surface Flattened for Determining Time of Setting by Gillmore Method.



(*b*) Gillmore Needles.

FIG. 4.

lb. In making the test, the needles shall be held in a vertical position and applied lightly to the surface of the pat.

TENSION TESTS

Form of Test Piece.

47. The form of test piece shown in Fig. 5 shall be used. The molds shall be made of non-corroding metal and have sufficient material in the sides to prevent spreading during molding. Gang molds when used shall be of the type shown in Fig. 6. Molds shall be oiled with a mineral oil.

Standard Sand.

48. The sand to be used shall be natural sand from Ottawa, Ill., screened to pass a No. 20 sieve and retained on a No. 30 sieve. This sand may be obtained from the Ottawa Silica Co., Ottawa, Ill.

49. This sand shall be considered standard when not more than 5 g. are retained on the No. 20 sieve, and not more than 5 g. pass the No. 30 sieve, after one minute continuous sieving of a 100-g. sample,

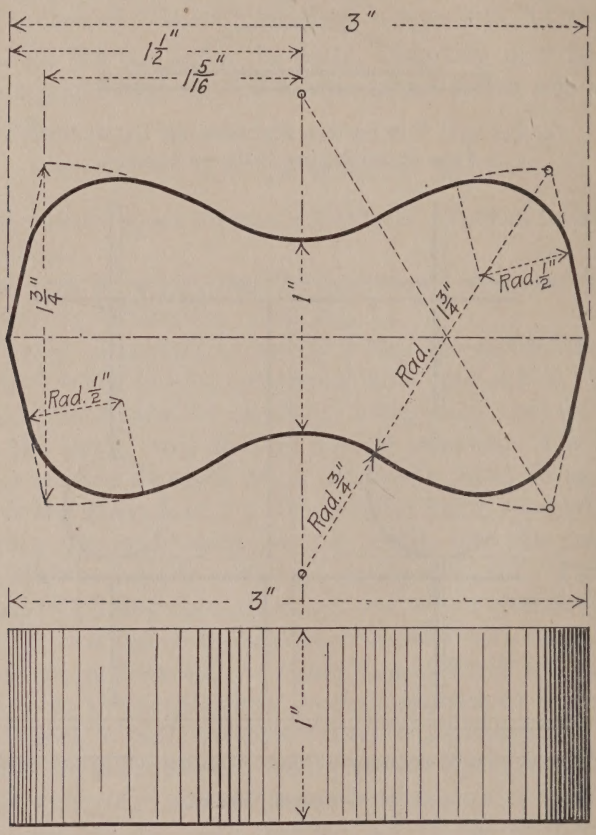


FIG. 5.—Details for Briquet.

in the manner specified for sieving cement on the No. 200 sieve (see Section 31).

50. The Nos. 20 and 30 sieves shall conform to the requirements for these sieves as given in the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials.[1]

Molding.

51. Immediately after mixing the standard mortar in accordance with the methods for mixing cement pastes and mortars the molds

[1] See p. 917.

shall be filled heaping full without compacting. Then the mortar shall be pressed in firmly with the thumbs, applying pressure 12 times to each briquet, at points to include the entire surface. The pressure shall be such that the simultaneous application of both thumbs will register a pressure of between 15 and 20 lb. Each application of the thumbs shall be maintained not longer than sufficient to attain the specified pressure. Then the mortar shall be heaped above the mold and smoothed off with a trowel. The trowel shall be drawn over the mold in such a manner as to exert a pressure of not more than 4 lb. The mold shall then be turned over upon a plane plate oiled with mineral oil, and the operation of heaping, thumbing and smoothing off repeated. No ramming or tamping shall be used, nor any troweling in excess of that required to smooth off the specimen.

Testing.

52. The briquets shall be tested as soon as they are removed from the water. Tests may be made with any machine meeting the following requirements: The machine shall be capable of weighing

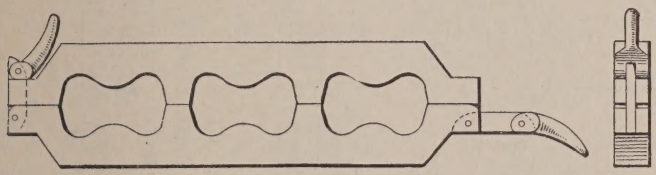


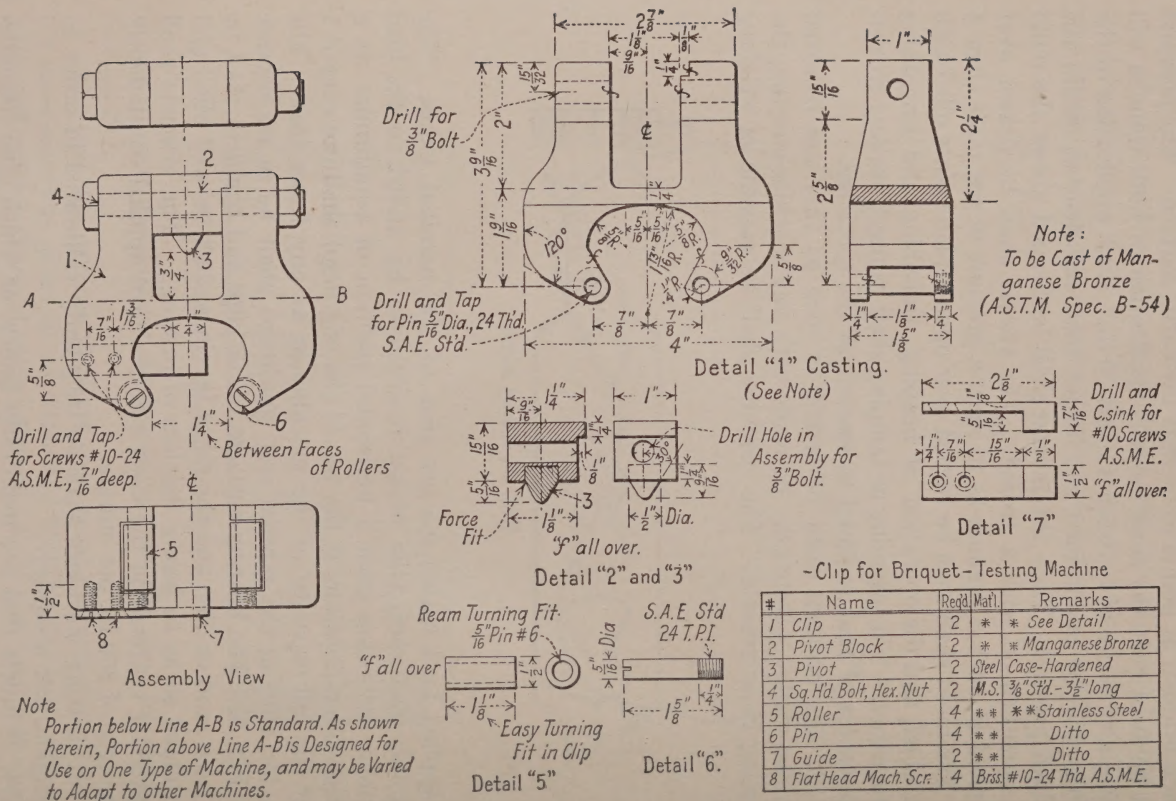
FIG. 6.—Gang Mold.

the applied load within 0.5 per cent of the nominal value. The sensibility reciprocal (the weight required to be added to the load to move the beam from a horizontal position of equilibrium to a position of equilibrium at the top of the trig loop) shall not exceed 1 lb. at the full capacity of the machine or at any lesser load. The clips for holding the tension test specimens shall be in accordance with Fig. 7. The bearing surfaces of the clips and briquets shall be free from sand or dirt, and the roller bearings shall be well oiled and maintained so as to insure freedom of turning. The briquets shall be carefully centered in the clips and the load applied continuously at the rate of 600 lb. per minute.

53. Testing machines shall be frequently calibrated in order to determine their accuracy.

Faulty Briquets.

54. Briquets that are manifestly faulty, or which give strengths differing more than 15 per cent from the average value of all test specimens made from the same sample and tested at the same period, shall not be considered in determining the tensile strength.


Drill for 3/8" Bolt
Drill and Tap for Pin 5/16" Dia., 24 Th'd S.A.E. St'd.
Detail "1" Casting.
(See Note)
Note: To be Cast of Manganese Bronze (A.S.T.M. Spec. B-54)
Drill and Tap for Screws #10-24 A.S.M.E., 7/16" deep.
Between Faces of Rollers
Assembly View
Drill Hole in Assembly for 3/8" Bolt.
Force Fit
"f" all over.
Detail "2" and "3"
Drill and C.sink for #10 Screws A.S.M.E.
"f" all over.
Detail "7"
Ream Turning Fit 5/16" Pin #6
S.A.E. Std 24 T.P.I.
"f" all over
Easy Turning Fit in Clip
Detail "5"
Detail "6"

Note
Portion below Line A-B is Standard. As shown herein, Portion above Line A-B is Designed for Use on One Type of Machine, and may be Varied to Adapt to other Machines.

-Clip for Briquet-Testing Machine

#	Name	Reqd.	Mat'l.	Remarks
1	Clip	2	*	* See Detail
2	Pivot Block	2	*	* Manganese Bronze
3	Pivot	2	Steel	Case-Hardened
4	Sq. Hd. Bolt, Hex. Nut	2	M.S.	3/8" Std. - 3 1/2" long
5	Roller	4	**	** Stainless Steel
6	Pin	4	**	Ditto
7	Guide	2	**	Ditto
8	Flat Head Mach. Scr.	4	Brass	#10-24 Th'd. A.S.M.E.

FIG. 7.—Clip for Briquet-Testing Machine.

STORAGE OF TEST SPECIMENS

55. The moist closet may consist of a soapstone, slate or concrete box, or a wooden box lined with metal. The interior wall surfaces of all closets shall be covered with felt or broad wicking kept wet. The bottom of the moist closet shall be covered with water to a depth of at least 2 in. **Apparatus.**

56. All test specimens, immediately after molding, shall be kept in the molds on plane plates in the moist closet for from 20 to 24 hours in such manner that the upper surfaces shall be exposed to the moist air. **Method.**

57. The briquets shall then be removed from the molds and immersed in clean water in storage tanks constructed of non-corroding material.

STANDARD SPECIFICATIONS
FOR
NATURAL CEMENT[1]

Serial Designation: C 10–09

These specifications are issued under the fixed designation C 10; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1904; REVISED, 1908, 1909.

Definition.

1. Natural cement is the finely pulverized product resulting from the calcination of an argillaceous limestone at a temperature only sufficient to drive off the carbonic acid gas.

PHYSICAL PROPERTIES

Fineness.

2. The residue on a standard No. 100 sieve[2] shall not exceed 10 per cent, and on a standard No. 200 sieve[2] shall not exceed 30 per cent, by weight.

Soundness.

3. Pats of neat cement about 3 in. in diameter, $\frac{1}{2}$ in. thick at center, tapering to a thin edge, shall be kept in moist air for a period of 24 hours.

(*a*) A pat shall then be kept in air at normal temperature.

(*b*) Another pat shall be kept in water maintained as near 70° F. as practicable.

These pats shall be observed at intervals for at least 28 days, and, to satisfactorily pass the tests, shall remain firm and hard and show no signs of distortion, checking, cracking, or disintegrating.

Time of Setting.

4. The cement shall not develop initial set in less than 10 minutes, using the Vicat needle. Final set shall be attained in not less than 30 minutes nor more than 3 hours, using the Vicat needle.

Tensile Strength.

5. The minimum requirements for tensile strength for briquets 1 sq. in. in cross-section shall be as follows, and the cement shall show no retrogression in strength within the periods specified:

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] For detailed specifications for these sieves, see the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, p. 917.

Neat Cement

AGE	STRENGTH
24 hours in moist air	75 lb.
7 days (1 day in moist air, 6 days in water)	150 "
28 days (1 " " " 27 " ")	250 "

One Part Cement, Three Parts Standard Ottawa Sand

7 days (1 day in moist air, 6 days in water)	50 lb.
28 days (1 " " " 27 " ")	125 "

PACKAGES, MARKING AND STORAGE

Packages and Marking.

6. The cement shall be delivered in suitable bags or barrels with the brand and name of the manufacturer plainly marked thereon. A bag shall contain 94 lb. net. A barrel shall contain 282 lb. net.

Storage.

7. The cement shall be stored in such a manner as to permit easy access for proper inspection and identification of each shipment, and in a suitable weather-tight building which will protect the cement from dampness.

INSPECTION

Inspection.

8. (*a*) Every facility shall be provided the purchaser for careful sampling and inspection at either the mill or at the site of the work, as may be specified by the purchaser. At least 10 days from the time of sampling shall be allowed for the completion of the 7-day test, and at least 31 days shall be allowed for the completion of the 28-day test.

(*b*) The cement shall be tested in accordance with the methods contained in the Standard Specifications and Tests for Portland Cement (Serial Designation: C 9) of the American Society for Testing Materials.[1]

REJECTION

Rejection.

9. The cement may be rejected if it fails to meet any of the requirements of these specifications.

10. Cement failing to meet the 7-day requirements may be held awaiting the results of the 28-day tests before rejection.

EDITORIAL NOTE

In connection with its reports on the Standard Specifications and Tests for Portland Cement (Serial Designation: C 9), Committee C-1 on Cement has declared its purpose to give consideration to the revision of the present Standard Specifications for Natural Cement, and recommended that in the meantime the methods of tests for Portland cement be applied to the testing of natural cement.

[1] See p. 23.

STANDARD SPECIFICATIONS

FOR

QUICKLIME FOR STRUCTURAL PURPOSES[1]

Serial Designation: C 5 – 26

These specifications are issued under the fixed designation C 5; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1915; REISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1926.

REQUIREMENTS

Requirements.

1. The quicklime shall conform to the following requirements as to chemical composition, calculated to the non-volatile basis:

	CALCIUM LIME	MAGNESIUM LIME
Calcium oxide, minimum, per cent	75	..
Magnesium oxide, minimum, per cent	..	20
Calcium and magnesium oxides, minimum, per cent	95	95
Silica, alumina, and oxide of iron, maximum, per cent	5	5
Carbon dioxide, maximum, per cent		
(*a*) If sample is taken at the kiln	3	3
(*b*) If sample is taken at any other place	10	10

Percentage of Residue.

2. When tested in accordance with the method described in Section 4, quicklime shall contain not more than 15 per cent by weight of residue.

METHODS OF TEST

Chemical Analysis.

3. The chemical analysis of the lime shall be made in accordance with the Tentative Methods of Chemical Analysis of Limestone, Quicklime and Hydrated Lime (Serial Designation: C 25 – 27 T) of the American Society for Testing Materials.[2]

Percentage of Residue.

4. An average $2\frac{1}{2}$-kg. (5-lb.) sample shall be put in a box of wood or of some material of similarly low thermal conductivity and slaked by an experienced operator with sufficient water at 70 to 80° F.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 194.

(21 to 27° C.) to produce the maximum quantity of lime putty, care being taken to avoid "burning" or "drowning" the lime. It shall be allowed to stand for 1 hour and then washed through a No. 20 sieve by a stream of water having a moderate pressure. No material shall be rubbed through the sieve. The washing shall be continued until the residue on the screen appears to consist wholly of coarse particles, but in no case shall washing be continued more than 30 minutes. The residue shall be dried to constant weight at a temperature of 212 to 225° F. (100 to 107° C.). The sample of lump lime taken for this test shall be broken so as to all pass a 1-in. ring and be retained on a $\frac{1}{4}$-in. sieve. Pulverized lime shall be tested as received.

SAMPLING, RETESTING, PACKING AND MARKING

Sampling, Retesting, Packing, Marking.

5. The sampling, retesting, packing and marking shall be conducted in accordance with the Standard Methods of Sampling, Inspection, Packing and Marking of Quicklime and Lime Products (Serial Designation: C 50) of the American Society for Testing Materials.[1]

APPENDIX

Quicklime can never be used as such for structural purposes; it must always be slaked first. Since the method of slaking is an important factor in determining the quality of the finished product, the following directions are given, not as a part of the specifications, but as information for the further protection of the purchaser.

PREPARATION OF LIME PUTTY

Directions for Slaking.

1. (*a*) *Introduction.*—Different kinds of lime vary considerably in the way in which they behave with water. A little supervision over the operation of slaking will amply pay for itself by insuring the production of the greatest possible quantity and the best possible quality of putty. To find out how to slake a new lot of lime, it is safest to try a little of it and see how it works. Since different lots of the same brand of lime vary somewhat, and since the weather conditions at the time have a decided influence, it is wise to try a sample from each lot used, whether familiar with the brand or not.

(*b*) *Classification of Limes.*—In a bucket, put two or three lumps of lime about the size of one's fist, or, in the case of granular lime, an equivalent amount. Add enough water to just barely cover the lime, and note how long it takes for slaking to begin. Slaking has begun when pieces split off from the lumps or when the lumps crumble. Water of the same temperature should be used for test and field practice.

If slaking begins in less than five minutes, the lime is quick slaking; from five to thirty minutes, medium slaking; over thirty minutes, slow slaking.

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[1] See p. 68.

(*c*) *Directions for Slaking.*—For quick-slaking lime, always add the lime to the water, not the water to the lime. Have enough water at first to cover all the lime completely. Have a plentiful supply of water available for immediate use—a hose throwing a good stream, if possible. Watch the lime constantly. At the slightest appearance of escaping steam, hoe thoroughly and quickly, and add enough water to stop the steaming. Do not be afraid of using too much water with this kind of lime.

For medium-slaking lime, add the water to the lime. Add enough water so that the lime is about half submerged. Hoe occasionally if steam starts to escape. Add a little water now and then if necessary to prevent the putty from becoming dry and crumbly. Be careful not to add any more water than required, and not too much at a time.

For slow-slaking lime, add enough water to the lime to moisten it thoroughly. Let it stand until the reaction has started. Cautiously add more water, a little at a time, taking care that the mass is not cooled by the fresh water. Do not hoe until the slaking is practically complete. If the weather is very cold, it is preferable to use hot water, but if this is not available, the mortar box may be covered in some way to keep the heat in.

Preparation of Putty for Use.

2. (*a*) *White Coat.*—After the action has ceased, run off the putty through a No. 10 sieve and store for a minimum of two weeks.

(*b*) *Base Coats.*—After the action has ceased, run off the putty through a No. 8 sieve. Add sand up to equal parts by weight, all of the hair required, and store for a minimum of two weeks.

(*c*) *Masons' Mortar.*—After the action has ceased, add part or all of the sand required, and store for a minimum of 24 hours.

STANDARD SPECIFICATIONS

FOR

HYDRATED LIME FOR STRUCTURAL PURPOSES[1]

Serial Designation: C 6 – 24

These specifications are issued under the fixed designation C 6; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1915; REISSUED AS TENTATIVE, 1917; ADOPTED IN AMENDED FORM, 1924.

Scope.

1. These specifications cover two classes of hydrated lime: namely, masons' hydrate and finishing hydrate.

Uses.

2. (*a*) Masons' hydrated lime may be used for scratch or brown coat of plaster, for stucco, for mortar, and for addition to portland-cement concrete.

(*b*) Finishing hydrated lime may be used for any of the purposes enumerated in Paragraph (*a*) for masons' hydrated lime, and in addition it may be used as an ingredient in the final or white coat of plaster.

REQUIREMENTS

Masons' Hydrate.

3. Masons' hydrated lime shall conform to the chemical and physical requirements herein specified except that for plasticity, specified in Section 8.

Finishing Hydrate.

4. Finishing hydrated lime shall conform to all chemical and physical requirements herein specified including that for plasticity, specified in Section 8.

Chemical Properties.

5. (*a*) *Carbon dioxide.*—Carbon dioxide in the sample shall not exceed 5 per cent if the sample is taken at the place of manufacture, or 7 per cent if the sample is taken at any other place. The percentages in both cases are based on the non-volatile portion. See Section 9.

(*b*) *Calcium and Magnesium Oxides.*—Calcium and magnesium oxides shall constitute not less than 95 per cent of the non-volatile portion. See Section 9.

Fineness.

6. The sample shall leave a residue of not more than 0.5 per cent on a No. 30 sieve, and not more than 15 per cent on a No. 200 sieve. See Section 10.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

Soundness. 7. If the steam has no visible effect on the pat (see Section 11), the sample shall be reported as being "sound." If the pat disintegrates, the sample shall be reported as being unsound, and the shipment rejected. If the pat cracks, pops, or shows other minor defects, it shall not be reported as either sound or unsound, but its behavior shall be noted.

Plasticity. 8. Finishing hydrated lime shall have a plasticity figure of not less than 200. See Section 13.

METHODS OF TEST

Chemical Analysis. 9. The chemical analysis of the hydrated lime shall be made in accordance with the Tentative Methods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime (Serial Designation: C 25 – 27 T) of the American Society for Testing Materials.[1]

Fineness. 10. Fineness of hydrated lime shall be determined as follows:

One hundred grams of the sample as received shall be placed on a No. 30 sieve which shall be nested above a No. 200 sieve. The material shall be washed by means of a stream of water from a faucet. The washing shall be continued until the water coming through the sieve is clear, but in no case should the washing be continued for more than 30 minutes. The residues on both sieves shall be dried to constant weight in an atmosphere free from carbon dioxide at a temperature between 100 and 120° C. The weights shall be calculated to percentages of the original sample, the weight of the material retained on the No. 30 sieve being added to the weight of the material retained on the No. 200 sieve to obtain the correct weight of the material retained on the No. 200 sieve.

The sieves used shall conform to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials.[2]

A piece of rubber tubing attached to a water faucet is to be used for the washing. The velocity of the water may be increased by pinching the tubing, but should not be sufficient to cause any splashing of the sample over the sides of the sieve.

Care must be taken not to let water accumulate on the No. 200 sieve, else the openings will become clogged and the operation cannot be completed in 30 minutes.

Constancy of Volume. 11. Constancy of volume shall be determined as follows:

To 20 g. of the sample, 100 g. of standard Ottawa sand shall be added, mixed thoroughly and enough water added to the mixture to

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 194.

[2] See p. 917.

make a good plastic mortar of rather dry consistency. This shall be spread on a clean glass plate to form a layer about $\frac{1}{4}$ in. thick by 4 in. square. The pat shall be of even thickness throughout, and not tapering at the edges. If the mortar is too dry to work well, more water may be added. The pat shall be placed in a closet for 24 hours. The temperature in the closet shall be between 65 and 75° F. (18 and 24°C.), and there shall be free circulation of air, but no direct draft striking the pat. The pat shall then be soaked in water until a film of water stands unabsorbed on the surface of the pat. If the pat has cracked, the consistency of the mortar was too wet and a new pat must be made. Two or three pats made at the same time from mortars of slightly different consistencies will avoid delays.

Twenty grams of the sample shall be mixed with enough water to form a thick cream. This shall be spread in a thin layer on the surface of the pat. It shall be allowed to stand for 15 minutes to permit air bubbles to form. It shall be troweled to an even surface, making this skim coat as thin as possible without allowing the sand to show through. The pat shall be put in the closet, under the conditions specified above, for 24 hours. It shall be examined carefully to see that there are no cracks or pops. The pat shall be suspended in a vessel partially filled with cold water, in such a way that the water can boil without touching the pat. The water shall be brought gradually to boiling and kept boiling gently for 5 hours, the pat being surrounded by steam during this time. The water shall then be permitted to cool for at least 12 hours. The pat shall then be removed and examined.

12. Consistency of lime putty shall be measured as follows: **Consistency.**

The lime shall be made into a stiff putty with water and permitted to soak over night. It shall be molded in a ring such as is used with a Vicat needle, resting the specimen on a glass plate.

The needle used is a modified form of Vicat needle, 12.5 mm. in diameter and weighing 30 g. This may conveniently be made from a suitable piece of aluminum tubing. The lower end should be closed without shoulders or curvature, and the tube loaded with shot to the specified weight. It is mounted in the Vicat needle stand.

The initial reading shall be taken with the bottom of the needle in contact with the surface of the sample. The final reading shall be taken 30 seconds after the plunger is released. A penetration of 20 mm. shall be considered standard.

If the penetration is less than standard, the sample may be removed from the mold, mixed with more water, and retested. If

the penetration is more than standard, the sample shall be discarded and a new one prepared.

The ring and the Vicat needle stand are described in the Standard Specifications and Tests for Portland Cement (Serial Designation: C 9) of the American Society for Testing Materials.[1]

Plasticity. 13. Plasticity shall be determined by means of the apparatus shown in Fig. 1 as follows: Three hundred grams of the sample shall be mixed with enough water to form a thick putty. After aging in a

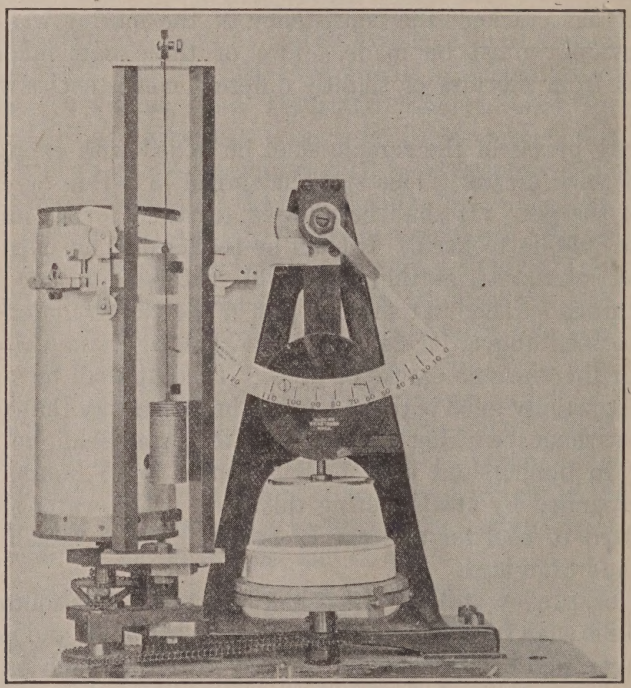

FIG. 1.—Emley Plasticimeter.

Constants of the Machine:

Absorption of porcelain base plate.—20 to 25 per cent.
Dimension of base plate.—1 in. in thickness by 4 in. in diameter.
Dimensions of disk.—$\frac{1}{32}$ in. in thickness by 3 in. in diameter.
Speed of vertical shaft.—One revolution in 6 minutes, 40 seconds.
Upward movement of base plate.—$\frac{1}{18}$ in. per revolution.
Torque on disk when bob reading is 100.—14,400 g.c.m.

vessel covered with a damp cloth for not less than 16 nor more than 24 hours, this putty shall be adjusted to standard consistency, as defined in Section 12, with a permissible variation of ±5 mm.

[1] See p. 23.

A ring such as is referred to in Section 12 shall be lubricated with a thin film of water, placed on a porcelain base plate, filled with the paste and struck off level. The mold shall be removed by raising it vertically without distorting the paste. The base plate and paste shall be placed in the instrument and the carriage turned up by hand until the surface of the paste is in contact with the disk and the distance between the disk and the top of the base plate is $1\frac{1}{4}$ in.

The carriage shall be thrown into gear and the motor started. It is essential that the motor shall be started exactly 120 seconds after the first portion of the paste has been put in the mold. The time when the first portion of paste is put in the mold shall be recorded as zero time—the motor is therefore started at two minutes.

Care shall be taken to protect the specimen from drafts during the test.

The scale reading shall be recorded every minute until the test is completed.

The test shall be considered complete when (*a*) the scale reading reaches 100; (*b*) any reading is less than the one before; or (*c*) the scale reading remains constant for three consecutive readings (two minutes) and the specimen has visibly ruptured or broken loose from the base plate.

The time and the scale reading at the end of the experiment shall be noted. The plasticity figure shall be calculated from the formula $P = \sqrt{F^2 + (10T)^2}$, in which P is the plasticity figure, F is the scale reading at the end of the experiment, and T is the time in minutes from when the first portion of paste was put in the mold to the end of the experiment.

After each test, the porcelain base plate shall be washed with hot water, treated with dilute hydrochloric acid to remove any lime from the surface pores, washed again with water, to remove the acid, dried by heating above 100° C., and cooled to room temperature before it is used again.

SAMPLING, INSPECTION, REJECTION, ETC.

14. The sampling, retesting, packing and marking shall be conducted in accordance with the Standard Methods of Sampling, Inspection, Packing and Marking of Quicklime and Lime Products (Serial Designation: C 50) of the American Society for Testing Materials.[1]

Sampling, Retesting, Packing, Marking.

[1] See p. 68.

STANDARD SPECIFICATIONS

FOR

QUICKLIME AND HYDRATED LIME FOR USE IN THE COOKING OF RAGS FOR THE MANUFACTURE OF PAPER[1]

Serial Designation; C 45 – 25

These specifications are issued under the fixed designation C 45; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED IN AMENDED FORM, 1925.

Lime in the Cooking of Rags.

1. Rags are cooked for the manufacture of paper in a digester under steam pressure with lime or with lime and soda-ash. They are then washed to eliminate as much of the non-cellulose material as possible.

REQUIREMENTS

Requirements.

2. (*a*) Either quicklime or hydrated lime may be used. It shall be clean and free from gritty substances.

(*b*) The standard quicklime for use in the cooking of rags for the manufacture of paper shall contain 90 per cent of available lime calculated on the basis of the weight of the sample taken at the point of manufacture (Note).

(*c*) The standard hydrated lime for use in the cooking of rags for the manufacture of paper shall contain 64.3 per cent of available lime[2] calculated on the basis of the weight of the sample taken at the point of manufacture (Note).

METHODS OF TEST

Chemical Analysis.

3. The chemical analysis for available lime shall be made in accordance with the Tentative Methods of Chemical Analysis of Limestone, Quicklime and Hydrated Lime (Serial Designation: C 25 – 27 T) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

[2] Available Lime of 64.3 per cent is equivalent to 85 per cent of available calcium hydroxide.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 194.

SAMPLING, INSPECTION, REJECTION, ETC.

4. The sampling, inspection, rejection, retesting, packing and marking shall be conducted in accordance with the Standard Methods of Sampling, Inspection, Packing and Marking of Quicklime and Lime Products (Serial Designation: C 50) of the American Society for Testing Materials.[1]

Sampling, Inspection, Rejection, etc.

EXPLANATORY NOTE.

A standard composition without rejection limits is specified for the reason that lime of either higher or lower total oxides, available lime, calcium oxide or calcium hydroxide, than the standard may safely be used under suitable conditions for the purpose herein specified depending primarily upon purely economic considerations. In the present state of the art it is believed that the more serviceable type of specification for the product herein specified is that which defines a reasonable standard rather than one that fixes actual rejection limits. It is generally recognized that, other things being equal, lime meeting this standard is preferable to lime not meeting it and lime surpassing the standard should be considered at a premium.

[1] See p. 68.

STANDARD SPECIFICATIONS

FOR

QUICKLIME FOR USE IN THE MANUFACTURE OF SULFITE PULP[1]

Serial Designation: C 46 – 27

These specifications are issued under the fixed designation C 46; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED IN AMENDED FORM, 1927.

Lime in Manufacture of Sulfite Pulp.

1. Lime is used in the "Milk of Lime" or "Tank System" of sulfite pulp manufacture for making the cooking liquor. The milk of lime is held in solution or suspension in a series of tanks equipped with suitable agitators. The sulfur dioxide is forced or drawn through these tanks successively. In some cases, the tanks are built on top of each other in the form of a tower. The contents of the first tank are drawn off when the liquor has reached a certain strength (3.5 to 6 per cent total SO_2) and the contents of the second and third tanks progress to the first and second tanks respectively. The third tank is again charged with fresh milk of lime. There are other systems of absorption which provide for continuous instead of intermittent operation. The function of the lime is to furnish the base for the formation of the bisulfites of calcium and magnesium.

REQUIREMENTS

Requirements.

2. (*a*) Either calcium or magnesium lime may be used, but owing to the greater solubility and reactivity of magnesium bisulfite, high magnesium lime gives the best results. The ratio of MgO to CaO shall be reasonably constant and the lime shall be moderately free from impurities.

(*b*) The quicklime shall conform to the following requirements

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

as to chemical composition, all percentages to be calculated on the basis of the weight of the sample taken at the point of manufacture:[1]

	CALCIUM LIME		MAGNESIUM LIME	
	MAXIMUM	MINIMUM	MAXIMUM	MINIMUM
Calcium oxide, per cent	....	92.5	....	55.4
Magnesium oxide, per cent	2.5	....	....	39.6
Oxides of silicon, iron and aluminum, per cent	3.0	....	3.0	....

METHODS OF TEST

Chemical Analysis.

3. The chemical analysis of the lime shall be made in accordance with the Tentative Methods of Chemical Analysis of Limestone, Quicklime and Hydrated Lime (Serial Designation: C 25 – 27 T) of the American Society for Testing Materials.[2]

SAMPLING, INSPECTION, REJECTION, ETC.

Sampling, Inspection, Rejection, etc.

4. The sampling, inspection, rejection, retesting, packing and marking shall be conducted in accordance with the Standard Methods of Sampling, Inspection, Packing and Marking of Quicklime and Lime Products (Serial Designation: C 50) of the American Society for Testing Materials.[3]

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[1] If it is desired to use the non-volatile basis the figures given above should be changed to the following:

	CALCIUM LIME		MAGNESIUM LIME	
	MAXIMUM	MINIMUM	MAXIMUM	MINIMUM
Calcium oxide, per cent	....	94.3	....	56.5
Magnesium oxide, per cent	2.6	....	....	40.4
Oxides of silicon, iron and aluminum, per cent	3.1	....	3.1	....

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 194.

[3] See p. 68.

STANDARD SPECIFICATIONS FOR HYDRATED LIME FOR THE MANUFACTURE OF VARNISH[1]

Serial Designation: C 47 – 27

These specifications are issued under the fixed designation C 47; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED, 1927.

Lime in the Manufacture of Varnish.

1. Hydrated lime is used in the manufacture of oleo-resinous varnishes to harden and partially neutralize the rosin. About 8 lb. of hydrated lime are gradually added to 100 lb. of the melted rosin, and heated for a short period.

REQUIREMENTS

Requirements.

2. (*a*) The hydrated lime shall be of such a color that no pronounced darkening of the finished varnish will result. It shall be of such a texture and of such a fineness that all portions of it will rapidly come into intimate contact with the resins so as to enter into the chemical reactions with rapidity.

(*b*) The lime shall be completely hydrated[2] since a high degree of hydration is important.

(*c*) *Fineness.*—Not more than 10 per cent of the hydrated lime shall be retained on a No. 230 (62-micron) sieve.

(*d*) *Reactivity.*—The hydrated lime shall conform to the requirements of the tung-oil heat test as indicated in Section 5.

(*e*) The hydrated lime shall conform to the following requirements as to chemical composition, calculated to the non-volatile basis:

Calcium oxide (CaO), minimum	94.0	per cent
Magnesium oxide (MgO), maximum	3.0	"
Iron oxide, alumina oxide and insoluble matter, maximum	3.0	"
Iron oxide (Fe_2O_3), maximum	0.4	"

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

[2] A very high calcium lime is preferred. It is especially important that the iron content be low in order to avoid undesirable color effects.

Carbon dioxide (CO_2), maximum:

(*a*) If sample is taken at place of manufacture....	3.0 per cent
(*b*) If sample is taken at other than place of manufacture.....	5.0 "

METHODS OF TEST

Chemical Analysis.

3. The chemical analysis of the lime shall be made in accordance with the Tentative Methods of Chemical Analysis of Limestone, Quicklime and Hydrated Lime (Serial Designation: C 25 – 27 T) of the American Society for Testing Materials.[1]

Fineness.

4. One hundred grams of the sample as received shall be placed on a No. 230 (62-micron) sieve.[2] The material shall be washed by means of a stream of water from a faucet (Note 1). Washing shall be continued until the water coming through the sieve is clear. The residue upon the No. 230 (62-micron) sieve shall be dried to constant weight in an atmosphere free from CO_2 in a drying oven whose temperature is maintained between 100 and 120° C. (212 and 248° F.). The weight of this residue shall be calculated as percentage of the original sample.

NOTE 1.—A small piece of rubber tubing attached to a water faucet will be found convenient. The velocity of the stream of water may be increased by pinching the tube, but the velocity should not be sufficient to cause any danger of splashing the sample over the sides of the sieve.

Tung Oil Heat Test.

5. Fifty grams of raw tung oil[3] shall be placed in a 100-cc. beaker and 1 g. of hydrated lime stirred in. A cloudy mass will result. Heat shall then be applied. High-quality hydrated lime will usually start to react at approximately 80° C. (176° F.), small bubbles rising to the surface. As heating is continued, large flakes of lime tungate will form and rise to the surface to form a thick white scum and the oil below will gradually become fairly clear. These changes will usually be completed when a temperature of 120° C. (248° F.) is reached. The heating shall be continued until the mass reaches a temperature of 140° C. (284° F.). It shall then be allowed to cool and the formation of the thick scum on the surface noted which will, when freed of air, gradually drop to the bottom of the beaker. This scum consists of

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 194.

[2] For detailed specifications for these sieves, see the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

[3] Tung oil for this test should conform to the requirements of the Tentative Specifications for Raw Tung Oil (Serial Designation: D 12 – 25 T) of the American Society for Testing Materials. See *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part I, p. 636 (1925); also 1927 Book of A.S.T.M. Tentative Standards, p. 304.

tung oil soaps of lime which are quite insoluble in the oil. Addition of rosin will readily effect solution of these soaps.

A poor grade of lime will fail to react, the absence of bubbles or flakes of lime tungate being noticeable. The oil will remain quiescent and cloudy. Only small amounts of soaps will form on the surface.

SAMPLING, INSPECTION, REJECTION, ETC.

Sampling, Inspection, Rejection, etc.

6. The sampling, inspection, rejection, retesting, packing and marking shall be conducted in accordance with the Standard Methods of Sampling, Inspection, Packing and Marking of Quicklime and Lime Products (Serial Designation: C 50) of the American Society for Testing Materials.[1]

[1] See p. 68.

STANDARD SPECIFICATIONS
FOR
QUICKLIME AND HYDRATED LIME FOR USE IN THE TEXTILE INDUSTRY[1]

Serial Designation: C 48 – 24

These specifications are issued under the fixed designation C 48; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED, 1924.

1. Lime is used in the textile industry for the following operations: **Lime in Textile Industry.**
   (*a*) Liming the kiers.
   (*b*) Boiling out or scouring.
   (*c*) Bleaching.
   (*d*) Mercerizing.
   (*e*) Water Softening.

These specifications apply only to the first three operations[2] in connection with the treatment of the vegetable fibers: cotton, linen and jute. For specifications for lime for water softening, see the Standard Specifications for Quicklime for Use in Water Treatment (Serial Designation: C 53) and the Standard Specifications for Hydrated Lime for Use in Water Treatment (Serial Designation: C 54) of the American Society for Testing Materials.[3]

2. (*a*) Kier liming consists of applying a coating of whitewash to the inside of the kier and heating with steam. These two operations are repeated until a coat of the desired thickness is obtained. The lime serves to prevent the fibers from being stained by the iron kier.

(*b*) The boiling-out process consists of soaking the fibers in a milk of lime, passing them through rollers into a kier, covering the fibers with water and boiling with steam either at atmospheric or higher pressures. The fibers then pass through a series of washings and treatments with acid and soda-ash. The function of the lime is to saponify, hydrolyze and neutralize whereby it makes possible, in

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

[2] Work is in progress on separate specifications for causticization (which is involved in mercerization).

[3] See pp. 64 and 66.

following operations, the removal of fatty and greasy matters and pectin bodies. The fibers then consist of practically pure cellulose, the impurities being coloring matter not decomposed by alkalis.

(*c*) Lime is used in the bleaching operation for making the "chemic" solution or hypochlorite bath. This bath may be made from bleaching powder or from lime and liquid chlorine. These specifications are limited to the process involving the use of lime and liquid chlorine. The operation consists of making a milk of lime and introducing chlorine under carefully controlled conditions until the mixture is nearly neutral. The sludge is allowed to settle and the clear hypochlorite solution is drawn off. After bleaching, the fibers are scoured and washed, and then passed on to the soda-ash boil. Lime serves as a carrier of chlorine under conditions whereby the liberation of nascent oxygen and the resulting oxidation can be controlled in accordance with the special requirements of textile fibers. Bleaching decolorizes the fibers by oxidation of the coloring matter. It probably performs several other functions also about which there is not general agreement.

REQUIREMENTS

Requirements.

3. (*a*) Either quicklime or hydrated lime of fairly high calcium content may be used.[1] The lime shall be fresh and substantially free from ash, dirt and core, and shall be very reactive chemically.

(*b*) The quicklime or hydrated lime shall conform to the following requirements as to chemical composition, calculated to the non-volatile basis:

Calcium oxide (CaO), minimum	94.0	per cent
Magnesium oxide (MgO), maximum	3.0	"
Iron and Aluminum oxides (Fe_2O_3 and Al_2O_3), maximum	2.0	"
Silica (SiO_2) and insoluble matter, maximum	2.5	"
Carbon dioxide (Co_2), maximum:		
(*a*) If sample is taken at place of manufacture		
(1) Quicklime	3.0	"
(2) Hydrated lime	5.0	"
(*b*) If sample is taken at other than place of manufacture		
(1) Quicklime	5.0	"
(2) Hydrated lime	7.0	"

METHODS OF TEST

Chemical Analysis.

4. The chemical analysis of the lime shall be made in accordance with the Tentative Methods of Chemical Analysis of Limestone, Quick-

[1] There is a tendency to prefer quicklime for the boiling out and kier liming process, and hydrated lime for the mordanting process. Quicklime or hydrated lime which contains iron in a readily soluble form should be avoided as should also hydrated lime which contains a considerable amount of unhydrated magnesia.

lime and Hydrated Lime (Serial Designation: C 25 – 27 T) of the American Society for Testing Materials.[1]

SAMPLING, INSPECTION, REJECTION, ETC.

5. The sampling, inspection, rejection, retesting, packing and marking shall be conducted in accordance with the Standard Methods of Sampling, Inspection, Packing and Marking of Quicklime and Lime Products (Serial Designation: C 50) of the American Society for Testing Materials.[2]

Sampling, Inspection, Rejection, etc.

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 194.

[2] See p. 68.

STANDARD SPECIFICATIONS
FOR
QUICKLIME AND HYDRATED LIME FOR THE MANUFACTURE OF SILICA BRICK[1]

Serial Designation: C 49 – 24

These specifications are issued under the fixed designation C 49; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED, 1924.

Lime in Manufacture of Silica Brick.

1. In the manufacture of silica brick, silica, preferably quartzite, is ground in a wet-pan until the particles are less than ¼ in. in size. Milk of lime is then added in quantities varying from 1.5 to 4 per cent CaO and the shapes are molded and dried. When bone-dry they are burned in down-draft kilns until most of the quartzite has been converted into tridymite and christobalite.

REQUIREMENTS

Requirements.

2. Either quicklime or hydrated lime may be used. It shall be substantially free from ash, dirt and core. Fineness of hydrate and rapidity of slaking of quicklime are desirable qualities.

(*b*) The quicklime or hydrated lime shall conform to the following requirements as to chemical composition, calculated to the non-volatile basis:

Calcium oxide (CaO), minimum	92.0	per cent
Magnesium oxide (MgO), maximum	3.0	"
Combined Iron and Aluminum oxides (Fe_2O_3 and Al_2O_3), maximum	1.5	"
Silica (SiO_2) and insoluble matter, maximum	3.0	"
Carbon dioxide (CO_2), maximum:		
(*a*) If sample is taken at place of manufacture	5.0	"
(*b*) If sample is taken at other than place of manufacture	10.0	"

METHODS OF TEST

Chemical Analysis.

3. The chemical analysis of the lime shall be made in accordance with the Tentative Methods of Chemical Analysis of Limestone,

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

Quicklime and Hydrated Lime (Serial Designation: C 25 – 27 T) of the American Society for Testing Materials.[1]

SAMPLING, INSPECTION, REJECTION, ETC.

4. The sampling, inspection, rejection, retesting, packing and marking shall be conducted in accordance with the Standard Methods of Sampling, Inspection, Packing and Marking of Quicklime and Lime Products (Serial Designation: C 50) of the American Society for Testing Materials.[2]

Sampling, Inspection, Rejection, etc.

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 194.

[2] See p. 68.

STANDARD SPECIFICATIONS
FOR
QUICKLIME FOR USE IN WATER TREATMENT[1]

Serial Designation: C 53 – 27

These specifications are issued under the fixed designation C 53; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1924; ADOPTED IN AMENDED FORM, 1927.

Quicklime for Water Treatment.

1. (*a*) In the treatment of water for public supplies, lime is used alone or with iron sulfate or aluminum sulfate to produce a precipitate which assists in the clarification of the water and in the removal of the bacteria by filtration. An excess of lime is sometimes used to remove part of the hardness of the water.

(*b*) Lime and soda ash are used together for softening water.

(*c*) The lime serves as a chemical reagent in water treatment. The only useful constituent of the lime is the calcium oxide capable of reacting with the other chemicals which are in the water or which are added to it. Inert material, besides reducing the value in proportion to its amount, also makes more sludge to be disposed of for a given amount of chemical action and thus reduces the capacity of the equipment in which it is used.

REQUIREMENTS

Requirements.

2. (*a*) The lime shall be substantially free of core, ash, and dirt, and shall be capable of disintegrating, in water, into a suspension of finely divided material.

(*b*) The standard quicklime for use in water treatment shall contain 90 per cent of available lime calculated on the basis of the weight of the sample taken at the point of manufacture. (Note).

METHODS OF TEST

Chemical Analysis.

3. The chemical analysis for available lime shall be made in accordance with the Tentative Methods of Chemical Analysis of

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

Limestone, Quicklime and Hydrated Lime (Serial Designation: C 25 – 27 T) of the American Society for Testing Materials.[1]

SAMPLING, INSPECTION, REJECTION, ETC.

4. The sampling, inspection, rejection, retesting, packing and marking shall be conducted in accordance with the Standard Methods of Sampling, Inspection, Packing and Marking of Quicklime and Lime Products (Serial Designation: C 50) of the American Society for Testing Materials.[2]

Sampling, Inspection Rejection, etc.

EXPLANATORY NOTE

A standard composition without rejection limits is specified for the reason that lime of either higher or lower total oxides, available lime, calcium oxide or calcium hydroxide, than the standard may safely be used under suitable conditions for the purpose herein specified depending primarily upon purely economic considerations. In the present state of the art it is believed that the more serviceable type of specification for the product herein specified is that which defines a reasonable standard rather than one that fixes actual rejection limits. It is generally recognized that, other things being equal, lime meeting this standard is preferable to lime not meeting it and lime surpassing the standard should be considered at a premium.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 194.

[2] See p. 68.

STANDARD SPECIFICATIONS

FOR

HYDRATED LIME FOR USE IN WATER TREATMENT[1]

Serial Designation: C 54 – 27

These specifications are issued under the fixed designation C 54; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1924; ADOPTED IN AMENDED FORM, 1927.

Hydrated Lime for Water Treatment.

1. (*a*) In the treatment of water for public supplies, lime is used alone or with iron sulfate or aluminum sulfate to produce a precipitate which assists in the clarification of the water and in the removal of bacteria by filtration. An excess of lime is sometimes used to remove part of the hardness of the water.

(*b*) Lime and soda ash are used for softening water.

(*c*) Hydrated lime serves as a chemical reagent in water treatment. The only useful constituent of the hydrated lime is the calcium hydroxide capable of reacting with the other chemicals which are in the water or which are added to it. Inert material, besides reducing the value in proportion to its amount, also makes more sludge to be disposed of for a given amount of chemical action and thus reduces the capacity of the equipment in which it is used.

REQUIREMENTS

Requirements.

2. The standard hydrated lime for use in water treatment shall contain 68.1 per cent of available lime[2] calculated on the basis of the weight of the sample taken at the point of manufacture (Note).

METHODS OF TEST

Chemical Analysis.

3. The chemical analysis for available lime shall be made in accordance with the Tentative Methods of Chemical Analysis of

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

[2] Available lime of 68.1 per cent is equivalent to 90 per cent of calcium hydroxide.

Limestone, Quicklime and Hydrated Lime (Serial Designation: C 25 – 27 T) of the American Society for Testing Materials.[1]

SAMPLING, INSPECTION, REJECTION, ETC.

4. The sampling, inspection, rejection, retesting, packing and marking shall be conducted in accordance with the Standard Methods of Sampling, Inspection, Packing and Marking of Quicklime and Lime Products (Serial Designation: C 50) of the American Society for Testing Materials.[2]

Sampling, Inspection, Rejection, etc.

EXPLANATORY NOTE

A standard composition without rejection limits is specified for the reason that lime of either higher or lower total oxides, available lime, calcium oxide or calcium hydroxide, than the standard may safely be used under suitable conditions for the purpose herein specified depending primarily upon purely economic considerations. In the present state of the art it is believed that the more serviceable type of specification for the product herein specified is that which defines a reasonable standard rather than one that fixes actual rejection limits. It is generally recognized that, other things being equal, lime meeting this standard is preferable to lime not meeting it and lime surpassing the standard should be considered at a premium.

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 194.

[2] See p. 68.

STANDARD METHODS OF SAMPLING, INSPECTION, PACKING AND MARKING OF QUICKLIME AND LIME PRODUCTS[1]

Serial Designation: C 50 - 27

These specifications are issued under the fixed designation C 50; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED IN AMENDED FORM, 1927.

METHODS OF SAMPLING

A. General

General.

1. (*a*) Samples of quicklime and lime products shall be taken at one of the following places, as may be agreed upon by the contracting parties:

At point of manufacture, or destination: From conveyors delivering to bins or piles, from filled bins by means of sampling tubes, from filled bins at the point of discharge, from piles by means of sampling tubes or shovels, or from loaded cars.

(*b*) All samples shall be taken in triplicate and immediately sealed in air-tight moisture-proof containers.

(*c*) One sample shall be immediately delivered to the consignee; one sample shall be delivered to the consignor if requested within 10 days from the date of sampling; the third sample shall be retained with seal unbroken until such time as the material has been accepted or a referee test called for.

(*d*) Samples shall be of an equal weight of not less than 5 lb. each when the quantity sampled equals 30 tons or more. For smaller quantities, the weights of the samples may be reduced proportionately, provided that no sample shall weigh less than 2 lb., except by special agreement of the contracting parties.

(*e*) If the samples are taken at any place other than the place of manufacture of the material being sampled, such samples shall be taken within 24 hours of the receipt of the material.

(*f*) Sampling shall be conducted as expeditiously as possible in order to avoid undue exposure of the material to the air.

(*g*) Samples shall not be taken from the broken packages.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

B. Lump or Granular Material

2. (*a*) *In Bulk* (Piles or cars).—Material shall be selected so that it will represent an average of all parts of the pile or car and shall not contain a disproportionate share of the top or bottom layers. This material shall comprise at least 10 shovelfuls taken from different parts of the pile or car. The total material so taken shall weigh not less than 100 lb. for a 30-ton unit, with a proportionate quantity of material for multiples of this unit, provided that at no time shall the total amount of material so taken weigh less than 50 lb. It shall be broken, if necessary, to pass a 1-in. ring, thoroughly mixed, and quartered, and the triplicate samples for testing shall be taken from the material so drawn and prepared. **Lump or Granular Material in Bulk.**

(*b*) *In Bulk* (At conveyors to or from bins, piles or cars).—Material shall be taken at regular intervals during the entire time of movement of the mass being sampled. The quantity to be taken at each interval and the number of such intervals shall be so proportioned that the final quantity of material taken shall conform to the requirements of Paragraph (*a*).

3. *In Packages.*—At least 2 per cent of the packages shall be sampled, the packages to be taken from various parts of the material being sampled, and at no time shall less than 5 packages be sampled. The contents of each of the packages so taken shall be put in a pile, broken, if necessary, to pass a 1-in. ring, thoroughly mixed, and quartered, and the triplicate samples for testing shall be taken from the material so drawn and prepared. **Lump or Granular Material in Packages.**

C. Powdered Material

4. (*a*) *In Bulk* (Bins, piles or cars).—A sampling tube shall be used which takes a core not less than 1 in. in diameter, and which is of sufficient length to permit the taking of a core from the top to the bottom of the mass being sampled. Cores shall be taken from at least 10 separate parts of the mass. The cores so taken shall be thoroughly mixed and quartered and the triplicate samples for testing shall be taken from the material so drawn and prepared. A minimum quantity of 100 lb. shall be drawn for a unit of 30 tons of material with a proportionate quantity of material for multiples of this unit, provided that at no time shall less than 50 lb. be drawn as the original portion. **Powdered Material in Bulk.**

(*b*) *In Bulk* (At conveyors to or from bins, piles or cars, at packing machines, etc.)—Sampling at conveyors, packing machines, etc., shall conform to the provisions of Section 2 (*b*).

Powdered Material in Packages.

5. *In packages.*—A sampling tube shall be used which takes a core not less than ½ in. in diameter. The tube shall be so inserted into the package that it will take a core of the material being sampled from substantially the entire length of the package. Material thus taken from individual packages shall be thoroughly mixed and quartered, and the triplicate samples for testing shall be taken from the material so drawn and prepared. Individual packages shall be taken from various parts of the unit being sampled. At least 1.5 per cent of the packages shall be sampled, and in no case shall less than 5 packages be sampled.

D. Treatment of Sample at Laboratory

Sample for Chemical Analysis.

6. The sample as received at the laboratory shall be thoroughly mixed, and reduced in quantity by the usual methods of quartering to give a minimum portion of 75 g. This portion shall be ground, either by a suitable mechanical device, or by hand in an agate mortar, until it all passes a No. 100 (149-micron) sieve.[1] It shall then be thoroughly mixed, placed in an air-tight container, and used in the determination of the chemical composition.

Samples for Special Tests.

7. Samples to be used for such special tests as may be required by individual specifications shall be obtained by mixing and quartering the material remaining after removal of the 75-g. portion for chemical analysis. The quantity of sample and the screen size of the particles will depend upon the requirements of the individual specifications.

INSPECTION

Inspection.

8. When inspection and sampling are to be conducted at the point of manufacture, the manufacturer shall afford the inspector representing the purchaser all reasonable facilities for inspection and sampling, which shall be so conducted as not to interfere unnecessarily with the operation of the works. The duties of the inspector or sampler shall be confined exclusively to sampling and inspection of the finished material as ready for shipment, and shall not under any circumstances be construed to permit investigation of methods or processes of manufacture.

REJECTION

Rejection.

9. (*a*) Rejection of material based on failure to pass the tests prescribed in the specifications shall be reported within 10 days from the taking of samples, and the cause for rejection shall be stated.

(*b*) The samples which represent rejected material shall be

[1] For detailed specifications for this sieve see the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

kept in air-tight, moisture-proof containers for at least 5 days from the date of the original test report.

RETESTING

10. (*a*) Either of the contracting parties may make claim for a retest within five days of the date of the original test report. The expense of the retest shall be borne by the party demanding such retest. **Retesting.**

(*b*). Should the contracting parties be unable to reach a mutually satisfactory agreement based upon the results of the original test, the third sample of material shall be delivered unopened to a referee laboratory for test and the results of this referee test shall be binding on both parties.

PACKING

11. *Lump Lime.*—Lump lime may be shipped in bulk in carload lots, in wooden barrels or steel containers holding 180 lb. or 280 lb. net each, or in pasteboard cartons holding 90 lb. net each. **Packing.**

12. *Pulverized Lime.*—Pulverized lime may be shipped in bulk, in carload lots, or in cloth or paper bags.

13. *Hydrated Lime.*—Hydrated lime shall be shipped in cloth bags holding 100 lb. net each, or in paper bags holding 50 lb. net each.

MARKING

14. Each shipment of material shall carry with it some means of identification, stating the particular contract on which the purchase and shipment were made. **Marking.**

15. Each package shall have legibly marked thereon, the net weight of its contents, the name of the manufacturer, the place of manufacture, and the brand name, if any. This marking shall comply with all requirements of the Federal Lime Barrel Act, entitled "An Act to Standardize Lime Barrels (Public No. 228-Congress-S 5425)," and with such other federal or state laws as may be applicable to the shipment in question.

16. In addition to the above information, the following guarantee may be marked on each package or shipment: "Guaranteed by (name of manufacturer) to meet the specifications of the American Society for Testing Materials" for (name of material).

STANDARD SPECIFICATIONS
FOR
GYPSUM[1]

Serial Designation: C 22 – 25

These specifications are issued under the fixed designation C 22; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1919; ADOPTED IN AMENDED FORM, 1925.

MATERIALS AND STANDARDS

Testing.

1. The chemical and physical properties of gypsum shall be determined in accordance with the Standard Methods of Testing Gypsum and Gypsum Products (Serial Designation: C 26) of the American Society for Testing Materials.[2]

Definition.

2. (*a*) Pure gypsum is calcium sulfate combined in crystallin form with two molecules of water.

(*b*) Its chemical formula is $CaSO_4 \cdot 2H_2O$.

(*c*) No material may be considered gypsum within the meaning of these specifications which contains less than 64.5 per cent by weight of $CaSO_4 \cdot 2H_2O$.

Forms and Sizes.

3. Gypsum may be in any of the following forms and sizes:

(*a*) *Run-of-Mine Gypsum.*—The form in which it comes from the mine or quarry.

(*b*) *Crushed Gypsum.*—Run-of-mine gypsum further reduced so that all of it will pass a 3-in. (76-mm.) ring, and not more than 25 per cent shall pass a No. 100 sieve.[3]

(*c*) *Sized Gypsum.*—Shall all pass a $1\frac{1}{2}$-in. (38-mm.) ring, and not more than 10 per cent shall pass a $\frac{1}{4}$-in. sieve.

(*d*) *Ground Gypsum.*—Gypsum further reduced for specific uses. Ground gypsum may be in any of the following standard forms:

No. 1.—This material shall all pass a No. 14 sieve,[3] and not less than 85 per cent of it shall pass a No. 100 sieve.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

[2] See p. 94.

[3] For detailed specifications for these sieves, see the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, p. 917.

No. 2.—This material shall all pass a No. 14 sieve, and not less than 60 nor more than 85 per cent of it shall pass a No. 100 sieve.

No. 3.—This material shall all pass a No. 8 sieve,[1] and not less than 40 nor more than 60 per cent of it shall pass a No. 100 sieve.

No. 4.—This material shall all pass a No. 100 sieve.

Chemical Properties.

4. The chemical composition, within the limits prescribed by Section 2, shall be a matter of contract.

SAMPLING

Sampling from Bulk.

5. When gypsum is shipped in bulk, samples of about 5 lb. (2.25 kg.) each shall be taken at regular intervals during the complete loading or unloading of the carrier, in such a way as to accumulate at least 200 lb. (90 kg.) of material, and to represent correctly the percentages of both the coarse particles and the fines in the shipment. This shall be broken so that it will all pass a 1-in. (25-mm.) ring, thoroughly mixed, and reduced by quartering to provide not less than a 1-lb. (0.5-kg.) sample for the laboratory.

Sampling from Packages.

6. When gypsum is shipped in packages, at least 3 per cent of the packages shall be sampled. Samples shall be taken from both the surface and the center of the packages. These samples shall then be broken, mixed, and quartered as directed above.

Laboratory Samples.

7. All laboratory samples shall be placed immediately in an air-tight container and shipped to the laboratory for test.

PACKING AND MARKING

Packing and Marking.

8. (*a*) Gypsum may be shipped either in packages or in bulk.

(*b*) When shipped in packages for resale, the following information shall be legibly marked on each package or on a tag of suitable size attached thereto:

Name of manufacturer;
Form and size of material;
Chemical analysis of the material, giving the minimum percentages of CaO and SO_3;
Net weight of package.

(*c*) When shipped in bulk, a card containing the required information shall be conspicuously placed in the carrier.

[1] For detailed specifications for these sieves, see the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, p. 917.

INSPECTION AND REJECTION

Inspection. 9. Inspection may be made either at the point of shipment or at the point of delivery. The inspector representing the purchaser shall have free access to the carriers being loaded for shipment to the purchaser. He shall be afforded all reasonable facilities for inspection and sampling, which shall be so conducted as not to interfere unnecessarily with the loading of the carriers.

Rejection. 10. Any rejection shall be based upon the specific cause of failure to conform to the requirements of these specifications, and shall be reported within ten working days from the receipt of the shipment by the consignee.

Rehearing. 11. Claims for rehearing shall be valid only if made within 20 working days from receipt of notice of specific cause for rejection.

STANDARD SPECIFICATIONS
FOR
CALCINED GYPSUM [1]

Serial Designation: C 23 – 22

These specifications are issued under the fixed designation C 23; the final number indicates the year of original adoption as standard, or in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1919; ADOPTED IN AMENDED FORM, 1922.

MATERIALS AND STANDARDS

Testing. 1. The chemical and physical properties of calcined gypsum shall be determined in accordance with the Standard Methods of Testing Gypsum and Gypsum Products (Serial Designation: C 26) of the American Society for Testing Materials.[2]

Definition. 2. Calcined gypsum is the product resulting from the partial dehydration of gypsum by means of heat.

Sizes. 3. Calcined gypsum may be marketed in either of the following sizes:

No. 1.—Material of this size shall all pass a No. 14 sieve,[3] and not less than 75 per cent of it shall pass a No. 100 sieve.[3]

No. 2.—Material of this size shall all pass a No. 14 sieve, and not less than 40 or more than 75 per cent of it shall pass a No. 100 sieve.

NOTE.—A tolerance provision of 1 per cent on all sieve determinations is permitted. All sieve numbers refer to United States Standard Sieve Series.[3]

Fineness. 4. Calcined gypsum may be marketed in any of the sizes enumerated for calcined gypsum in Section 3. Each package or each shipment, shipped for resale, shall be accompanied by a tag or card which shall contain the information required by Section 10.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

[2] See p. 94.

[3] For detailed specifications for these sieves, see the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, p. 719.

Time of Setting.

5. (*a*) The time of set of calcined gypsum shall be suitable for the purposes intended, and shall conform to the provisions prescribed for such in the Standard Specifications for Gypsum Plasters (Serial Designation: C 28) of the American Society for Testing Materials.[1]

(*b*) Calcined gypsum for molding or casting shall set in not less than 10 minutes nor more than 40 minutes.

Tensile Strength.

6. (*a*) Calcined gypsum shall have a tensile strength of not less than 200 lb. per sq. in. (14 kg. per sq. cm.).

(*b*) Calcined gypsum for molding or casting shall have a tensile strength of not less than 200 lb. per sq. in. (14 kg. per sq. cm.).

Compressive Strength.

7. (*a*) Calcined gypsum shall have a compressive strength of not less than 1000 lb. per sq. in. (70 kg. per sq. cm.).

(*b*) Calcined gypsum for molding or casting shall have a compressive strength of not less than 1000 lb. per sq. in. (70 kg. per sq. cm.).

SAMPLING

Sampling.

8. At least 3 per cent of the packages shall be sampled, and shall be so selected as to be representative of the contents of the shipment. Samples shall be taken from both the surface and the center of the packages. The material so obtained shall be thoroughly mixed and reduced by quartering to provide not less than a 15-lb. (6.75 kg.) sample for the laboratory.

Laboratory Samples.

9. All laboratory samples shall immediately be placed in an air-tight container and shipped to the laboratory for test.

PACKING AND MARKING

Packing and Marking.

10. Calcined gypsum may be shipped in either packages or in bulk.

(*a*) When shipped for resale, the following information shall be legibly marked on each package or on a tag of suitable size attached thereto:

Name of manufacturer;
Brand;
Description;
Net weight of package.

(*b*) When shipped in bulk, a card containing the required information shall be conspicuously placed in the carrier.

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[1] See p. 78.

INSPECTION AND REJECTION

11. Inspection may be made either at the point of shipment or at the point of delivery. The inspector representing the purchaser shall have free access to the carriers being loaded for shipment to the purchaser. He shall be afforded all reasonable facilities for inspection and sampling, which shall be so conducted as not to interfere unnecessarily with the loading of the carriers. **Inspection.**

12. Any rejection shall be based upon the specific cause of failure to conform to the requirements of these specifications and shall be reported within ten working days from the receipt of the shipment by the consignee. **Rejection.**

13. Claims for rehearing shall be valid only if made within 20 working days from receipt of notice of specific cause for rejection. **Rehearing.**

STANDARD SPECIFICATIONS
FOR
GYPSUM PLASTERS[1]

Serial Designation: C 28 – 27

These specifications are issued under the fixed designation C 28; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1920; ADOPTED IN AMENDED FORM, 1921; REVISED, 1927.

Testing.

1. The chemical and physical properties of gypsum plasters shall be determined in accordance with the Standard Methods of Testing Gypsum and Gypsum Products (Serial Designation: C 26) of the American Society for Testing Materials.[2]

GYPSUM READY-SANDED PLASTER

Definition.

2. Gypsum ready-sanded plaster is a plastering material in which the predominating cementitious material is calcined gypsum, and which is mixed at the mill with all the constituent parts, including sand, in their proper proportion. It requires only the addition of water to make it ready for use.

Composition.

3. The following shall govern the composition of gypsum ready-sanded plaster for the desired coats:

(*a*) *Scratch or First Coat.*—Gypsum ready-sanded plaster used for scratch or first coat shall contain not more than two-thirds by weight of sand. The other 33 per cent shall contain not less than 60.5 per cent by weight of calcined gypsum, $CaSO_4 \cdot \frac{1}{2}H_2O$, calculated from the SO_3 content. The remainder may consist of materials to control the working quality, setting time, and the fibering.[3]

(*b*) *Browning or Second Coat.*—Gypsum ready-sanded plaster used for browning or second coat shall contain not more than 75 per cent by weight of sand. The other 25 per cent shall contain not less than 60.5 per cent by weight of calcined gypsum, $CaSO_4 \cdot \frac{1}{2}H_2O$, calculated from the SO_3 content. The remainder may consist of materials to control the working quality, setting time, and the fibering.[3]

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

[2] See p. 94.

[3] The U. S. Bureau of Standards is investigating the effect of portland cement mixed in gypsum plasters. A report on these investigations is forthcoming.

4. The time of set of gypsum ready-sanded plaster shall be as follows: **Time of Setting.**

(*a*) *Scratch or First Coat.*—This plaster shall set in not less than 1½ nor more than 7 hours.

(*b*) *Browning or Second Coat.*—This plaster shall set in not less than 2 nor more than 6 hours.

5. The tensile strength of gypsum ready-sanded plaster shall be as follows: **Tensile Strength.**

(*a*) *Scratch or First Coat.*—This plaster shall have a tensile strength of not less than 75 lb. per sq. in. (5¼ kg. per sq. cm.).

(*b*) *Browning or Second Coat.*—This plaster shall have a tensile strength of not less than 50 lb. per sq. in. (3½ kg. per sq. cm.).

GYPSUM NEAT PLASTER

6. Gypsum neat plaster is a plastering material in which not less than 60.5 per cent of the cementitious material is calcined gypsum, $CaSO_4 \cdot \frac{1}{2}H_2O$, calculated from the SO_3 content, and mixed at the mill with other materials. **Definition.**

7. Gypsum neat plaster shall contain not less than 60.5 per cent by weight of calcined gypsum, $CaSO_4 \cdot \frac{1}{2}H_2O$, calculated from the SO_3 content. The remainder may consist of materials to control the working quality, setting time, and the fibering.[1] **Composition.**

8. Gypsum neat plaster when mixed with three parts by weight of testing sand shall set in not less than 8 hours, nor more than 32 hours. **Time of Setting.**

9. Gypsum neat plaster shall have a tensile strength of not less than 150 lb. per sq. in. (10½ kg. per sq. cm.). **Tensile Strength.**

GYPSUM WOOD-FIBERED PLASTER

10. Gypsum wood-fibered plaster is a gypsum plaster in which wood fiber is used as an aggregate. **Definition.**

11. Gypsum wood-fibered plaster shall contain not less than 60.5 per cent by weight of calcined gypsum, $CaSO_4 \cdot \frac{1}{2}H_2O$, calculated from the SO_3 content, and not less than 1 per cent by weight of wood fiber made from a non-staining wood. The remainder may consist of materials to control the working quality and setting time.[1] **Composition.**

12. Gypsum wood-fibered plaster shall set in not less than 1½ nor more than 8 hours. **Time of Setting.**

13. Gypsum wood-fibered plaster shall have a tensile strength of not less than 125 lb. per sq. in. (8¾ kg. per sq. cm.). **Tensile Strength.**

[1] The U. S. Bureau of Standards is investigating the effect of portland cement mixed in gypsum plasters. A report on these investigations is forthcoming.

CALCINED GYPSUM FOR FINISHING COAT

Definition. 14. Calcined gypsum for finishing coat may, or may not, contain retarder. It may be classified in two grades: viz, "White" and "Gray."

Fineness. 15. Calcined gypsum for finishing coat shall all pass a No. 14 (1410-micron) sieve,[1] and not less than 60 per cent of it shall pass through a No. 100 (149-micron) sieve.[1]

Time of Setting. 16. Calcined gypsum for finishing coat when not retarded shall set in not less than 20 minutes nor more than 40 minutes, and when retarded shall set in not less than 40 minutes nor more than 6 hours.

Tensile Strength. 17. Calcined gypsum for finishing coat shall have a tensile strength of not less than 200 lb. per sq. in. (14 kg. per sq. cm.).

SAMPLING

Sampling. 18. At least 3 per cent of the packages shall be sampled, and shall be so selected as to be representative of the contents of the shipment. Samples shall be taken from both the surface and the center of the packages. The material so obtained shall be thoroughly mixed and reduced by quartering to provide not less than a 15-lb. (7.5-kg.) sample for the laboratory.

Laboratory Samples. 19. All laboratory samples shall immediately be placed in an air-tight container and shipped to the laboratory for test.

PACKING AND MARKING

Packing and Marking. 20. (*a*) Gypsum plasters shall be dry and free from lumps, and may be shipped either in packages or in bulk.

(*b*) When shipped for resale, the following information shall be legibly marked on each package or on a tag of suitable size attached thereto:

Name of Manufacturer.
Brand.
Description.
Net weight of package.

(*c*) When shipped in bulk, a card containing the required information shall be conspicuously placed in the carrier.

INSPECTION AND REJECTION

Inspection. 21. Inspection may be made either at the point of shipment or at the point of delivery. The inspector representing the purchaser

[1] For detailed specifications for these sieves, see the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, p. 917.

shall have free access to the carrier being loaded for shipment to the purchaser. He shall be afforded all reasonable facilities for inspection and sampling, which shall be so conducted as not to interfere unnecessarily with the loading of the carriers.

22. Any rejection shall be based upon the specific cause of failure to conform to the requirements of these specifications, and shall be reported within ten working days from the receipt of the shipment by the consignee. **Rejection.**

23. Claim for rehearing shall be valid only if made within twenty working days from receipt of notice of specific cause for rejection. **Rehearing.**

NOTE.

The water used for mixing gypsum plasters shall be fresh, pure and clean. Trouble may occur from the presence of any excessive amount of mineral or organic substances in the water.

STANDARD SPECIFICATIONS FOR GYPSUM PLASTERING SAND[1]

Serial Designation: C 35 – 25

These specifications are issued under the fixed designation C 35; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1925.

Composition. 1. Sand used for plastering in which gypsum is employed, shall consist of fine granular material, naturally or artifically produced by the disintegration of rock containing not less than 80 per cent by weight of silica, feldspar, dolomite, magnesite or calcite, and shall be free from saline, alkaline, organic or other deleterious substances.

Grading. 2. It shall be graded from fine to coarse, and when dry not more than 6 per cent by weight shall be retained on a No. 8 (2380-micron) sieve; not less than 80 per cent by weight shall be retained on a No. 50 (297 micron) sieve; and not more than 6 per cent by weight shall pass a No. 100 (149-micron) sieve. These sieves shall conform to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials.[2]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

[2] See p. 917.

STANDARD SPECIFICATIONS
FOR
GYPSUM WALL BOARD[1]

Serial Designation: C 36 – 25

These specifications are issued under the fixed designation C 36; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1925.

Definition. 1. Gypsum wall boards are sheets or slabs having an incombustible core of gypsum which, without the addition of plaster, furnish to interior walls, ceilings or partitions a surface which may receive decoration.

Composition. 2. Gypsum wall boards consist of an incombustible core of gypsum, with or without fiber, surfaced with paper or other fibrous material firmly bonded to the core.

Thickness. 3. Gypsum wall boards shall be nominally $\frac{3}{8}$ in. (9.5 mm.) in thickness with permissible variations in the average thickness of $\pm\frac{1}{64}$ in. (0.4 mm) and with permissible local variations of $\pm\frac{1}{32}$ in. (0.8 mm.) from the normal thickness.

NOTE.—Gypsum wall boards are manufactured in thicknesses from $\frac{1}{4}$ in. (6.3 mm.) to $\frac{1}{2}$ in. (12.7 mm.) with permissible local variations of $\pm\frac{1}{32}$ in. (0.8 mm.) from the nominal thickness.

Dimensions. 4. (*a*) Gypsum wall boards that are to be laid with the joints butted shall have widths of 32 in. (810 mm.), 36 in. (910 mm.), or 48 in. (1220 mm.), except that a variation of $\frac{3}{32}$ in. (2 mm.) under the specified widths is permissible.

(*b*) Gypsum wall boards that are to be laid with the joints filled shall have widths $\frac{1}{4}$ in. (6.3 mm.) less than those specified in Paragraph (*a*) except that the same variation is permissible.

(*c*) The lengths shall be from 4 ft. (1.22 meters) to 12 ft. (3.66 meters), inclusive, except that a tolerance of $\frac{3}{8}$ in. (9.5 mm.) from the length of individual boards is permissible. Special boards may be cut to any desired length within the limits prescribed.

Weight. 5. The weight of gypsum wall board shall be not less than 1400 lb. (635 kg.) nor more than 2100 lb. (952.5 kg.) per 1000 sq. ft.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

Strength. 6. (*a*) Strength test specimens shall be 12 in. (300 mm.) in width and approximately 18 in. (460 mm.) in length. When tested in accordance with the methods described in Section 13, specimens taken from the gypsum wall boards shall carry a load of not less than 60 lb. (27.22 kg.) when the load is applied across the fiber of the surfacing, and not less than 32 lb. (14.50 kg.) when the load is applied parallel to the fiber of the surfacing.

(*b*) With loads equal to or less than those specified in Paragraph (*a*) there shall be no breaking of the bond between the surfacing and the core.

Finish. 7. That surface of the gypsum wall board designed to be exposed on erection shall be true and free from imperfections that would render the wall boards unfit for use with or without decorations. The edges and ends shall be straight and solid. Boards that are to be butted shall have corners square and opposite edges parallel to each other. With boards that are to have the joints filled, a negative variation of $\frac{1}{8}$ in. (3 mm.) from parallelism of opposite edges is permissible.

Sampling. 8. At least one per cent of the number of gypsum wall boards in a shipment, but not less than ten boards, shall be so selected as to be representative of the shipment. Twenty-five per cent of such selected boards, but not less than ten individual boards, shall constitute a sample for test purposes. When a shipment consists of more than one car or carrier load, a sample shall be selected from each car or carrier.

Packing and Marking. 9. Shipments of gypsum wall board shall conform to the following:

(*a*) They shall be shipped so as to be kept dry and free from moisture.

(*b*) When shipped for resale the following information shall be shown upon each board:

Name of manufacturer;
Brand;
Nominal thickness.

Inspection. 10. Inspection may be made either at the point of shipment or at the point of delivery. The inspector representing the purchaser shall have free access to the carriers being loaded for shipment to the purchaser. He shall be afforded all reasonable facilities for inspection and sampling, which shall be so conducted as not to interfere unnecessarily with the loading of the carriers.

Rejection. 11. Any rejection shall be based upon failure to conform to the requirements of these specifications and shall be reported within ten working days from the receipt of the shipment by the consignee. The

notice of rejection shall contain a specific statement of the respects in which the boards have failed to meet the requirements of these specifications.

12. Claims for rehearing shall be valid only if made within twenty working days from the receipt of notice of specific cause for rejection. **Rehearing.**

METHOD OF TESTING

13. (*a*) Test specimens for the transverse strength test shall be taken from not less than ten gypsum boards. **Transverse Strength.**

(*b*) The test specimens shall be 12 in. (300 mm.) in width and approximately 18 in. (460 mm.) in length. They shall be supported on fixed parallel bearings spaced 16 in. (410 mm.) on centers, and shall be loaded through a similar bearing midway between the supports. All bearing and load surfaces shall be true, shall engage the full width of the test specimen, and shall be rounded to a radius of $\frac{1}{8}$ in. (3.2 mm.).

(*c*) Test loads shall be applied at a uniform rate of 60 lb. (27.22 kg.) per minute with a permissible variation of ± 10 per cent in the rate. Strength test results shall be reported when the load is applied across the fiber of the surfacing and also parallel to the fiber of the surfacing.

STANDARD SPECIFICATIONS
FOR
GYPSUM PLASTER BOARD[1]

Serial Designation: C 37 – 25

These specifications are issued under the fixed designation C 37; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1925.

Definition. 1. Gypsum plaster boards are sheets or slabs having an incombustible core of gypsum, and designed to be used as a lath or backing for gypsum plaster on the walls, ceilings and partitions in the interior of buildings.

Composition. 2. Gypsum plaster boards consist either of sheets or slabs of gypsum with not more than 15 per cent, by weight, of fiber intimately mixed; or an incombustible core of gypsum, with or without fiber, surfaced with paper or other fibrous material firmly bonded to the core, or with intermediate layers of such material within the core.

Thickness. 3. Gypsum plaster boards shall be $\frac{1}{4}$ in. (6.3 mm.), $\frac{5}{16}$ in. (8.0 mm.), $\frac{3}{8}$ in. (9.5 mm.), or $\frac{1}{2}$ in. (12.7 mm.) thick, except that average thicknesses varying not more than $\frac{1}{16}$ in. (1.6 mm.) from the nominal thicknesses, and local variations of $\frac{1}{16}$ in. (1.6 mm.) in the thickness of the individual boards are permissible.

Dimensions. 4. The nominal width of gypsum plaster boards shall be 32 in. (810 mm.), and the nominal length 24 in. (610 mm.), 36 in. (910 mm.) or 48 in. (1220 mm.). A variation of $\frac{1}{4}$ in. (6.3 mm.) under the specified width, and of $\frac{3}{8}$ in. (9.5 mm.) under or over the specified length is permissible.

Weight. 5. The weight of gypsum plaster board per 1000 sq. ft. shall conform to the following:

Thickness		Weight, Minimum		Weight, Maximum	
in.	mm.	lb.	kg.	lb.	kg.
$\frac{1}{4}$	6.3	1200	544	1500	680
$\frac{5}{16}$	8	1250	567	1650	750
$\frac{3}{8}$	9.5	1400	635	2000	907
$\frac{1}{2}$	12.7	2000	907	3000	1361

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

6. (*a*) Strength test specimens shall be 12 in. (300 mm.) wide and approximately 18 in. (460 mm.) long. When tested in accordance with the methods described in Section 13, specimens taken from the gypsum plaster boards shall carry not less than the following loads: **Strength.**

Board Thickness, in.	Load, lb. Knife Edges Across Fiber of Surfacing	Load, lb. Knife Edges Parallel to Fiber of Surfacing
$\frac{3}{8}$	40	20
$\frac{5}{16}$	35	17
$\frac{1}{4}$	30	14

(*b*) With loads equal to or less than those specified in Paragraph (*a*) there shall be no breaking of the bond between the surfacing and the core.

(*c*) Gypsum plaster boards that are $\frac{1}{2}$ in. (12.7 mm.) thick, or those that are not reinforced on the surfaces with a covering made from fibrous material, when tested as described in Paragraph (*a*), shall carry an ultimate center load of not less than 20 lb. (9.07 kg.).

7. The surfaces shall be such that they will readily receive and retain gypsum plaster. The edges and ends shall be reasonably straight and solid. The corners shall be square with a permissible variation of $\frac{1}{4}$ in. (6 mm.) in the full width of the plaster boards. Gypsum plaster boards shall be free from cracks and imperfections that will render them unfit for use. **Finish.**

NOTE.—Gypsum plaster boards that have their corners blurred or broken shall be considered acceptable, provided that the broken portion is not more than $1\frac{1}{2}$ in. (38 mm.) in length.

8. At least one per cent of the number of gypsum plaster boards in a shipment, but not less than ten boards, shall be so selected as to be representative of the shipment. Twenty-five per cent of such selected boards, but not less than ten individual boards, shall constitute a sample for test purposes. When a shipment consists of more than one car or carrier load, a sample shall be selected from each car or carrier. **Sampling.**

9. Shipments of gypsum plaster board shall conform to the following: **Packing and Marking.**

(*a*) They shall be shipped so as to be kept dry and free from moisture.

(*b*) When shipped for resale the following information shall be shown upon each board:

Name of manufacturer;
Brand;
Nominal thickness.

Inspection. 10. Inspection may be made either at the point of shipment or at the point of delivery. The inspector representing the purchaser shall have free access to the carriers being loaded for shipment to the purchaser. He shall be afforded all reasonable facilities for inspection and sampling, which shall be so conducted as not to interfere unnecessarily with the loading of the carriers.

Rejection. 11. Any rejection shall be based upon failure to conform to the requirements of these specifications, and shall be reported within ten working days from the receipt of the shipment by the consignee. The notice of rejection shall contain a specific statement of the respects in which the boards have failed to meet the requirements of these specifications.

Rehearing. 12. Claims for rehearing shall be valid only if made within twenty working days from the receipt of notice of specific cause for rejection.

METHOD OF TESTING

Transverse Strength. 13. (*a*) Test specimens for the transverse strength test shall be taken from not less than ten gypsum boards.

(*b*) The test specimens shall be 12 in. (300 mm.) in width and approximately 18 in. (460 mm.) in length. They shall be supported on fixed parallel bearings spaced 16 in. (410 mm.) on centers, and shall be loaded through a similar bearing midway between the supports. All bearing and load surfaces shall be true, shall engage the full width of the test specimen, and shall be rounded to a radius of $\frac{1}{8}$ in. (3.2 mm.).

(*c*) Test loads shall be applied at a uniform rate of 60 lb. (27.22 kg.) per minute with a permissible variation of ± 10 per cent in the rate. Strength test results shall be reported when the load is applied across the fiber of the surfacing and also parallel to the fiber of the surfacing.

STANDARD SPECIFICATIONS
FOR
GYPSUM PARTITION TILE OR BLOCK[1]

Serial Designation: C 52 – 27

These specifications are issued under the fixed designation C 52; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED IN AMENDED FORM, 1925; REVISED, 1927.

Definition. 1. Gypsum partition tile or block is a building unit used for non-bearing construction in the interior of buildings.

NOTE.—The words "tile" and "block" are synonymous and are used so as to be in accord with certain freight association nomenclature.

Uses. 2. (*a*) It is also used as a protection for columns, elevator shafts, etc., against fire.

(*b*) Gypsum cored tile, 3 or 4 in. in thickness, may be split and used for furring purposes.

MANUFACTURE

Composition. 3. Gypsum tile consists of gypsum with or without an aggregate.

DIMENSIONS AND PERMISSIBLE VARIATIONS

Form and Size. 4. Gypsum partition tile shall be rectangular in shape with straight and square edges and true surfaces. They may also be of special shape provided the requirements as otherwise mentioned herein, are met. They may be solid or cored and shall preferably be 12 by 30 in. in face dimensions.

Core Spaces. 5. (*a*) The core spaces shall be symmetrically spaced.

(*b*) *Shell Thickness.*—The minimum cross-sectional shell dimensions of gypsum partition tile with circular, elliptical or rectangular core spaces shall conform to the following:

Thickness of Tile or Block, in.	Side or Edge Shell Thickness, in.	
	Circular Core	Elliptical or Rectangular Core
$2\frac{1}{2}$	$\frac{1}{2}$	$\frac{9}{16}$
3	$\frac{1}{2}$	$\frac{11}{16}$
4	$\frac{3}{4}$	$\frac{13}{16}$
5	$\frac{7}{8}$	$1\frac{1}{8}$
6	$\frac{1}{2}$	$\frac{11}{16}$
8	$\frac{3}{4}$	$\frac{13}{16}$

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

In tile or block having circular cores, the sum of the widths of the two side shells plus the width of the center vertical web shall not be less than 2 in. in the case of 6-in. tile, and not less than $2\frac{1}{2}$ in. in the case of 8-in. tile.

In tile or block having elliptical or rectangular cores, the sum of the widths of the two side shells plus the width of the center vertical web shall be not less than $2\frac{3}{4}$ in. in the case of 6-in. tile, and not less than $3\frac{1}{4}$ in. in the case of 8-in. tile.

Dimensions.

6. (*a*) Gypsum tile may be of any convenient length and height, but in general they shall not exceed 30 in. in length and 12 in. in height. The thickness shall be uniform throughout and within the limits specified in Paragraph (*b*)

(*b*) *Thickness.*—Gypsum tile or block shall conform to the specified thicknesses with the following permissible variations:

Nominal Size of Tile or Block, in.	Minimum Thickness, in.	Maximum Thickness, in.
$1\frac{1}{2}$ (Furring)	$1\frac{3}{8}$	$1\frac{5}{8}$
2 (Furring)	$1\frac{7}{8}$	$2\frac{1}{8}$
2	2	$2\frac{1}{4}$
$2\frac{1}{2}$	$2\frac{3}{8}$	$2\frac{3}{4}$
3	$2\frac{7}{8}$	$3\frac{1}{4}$
4	$3\frac{7}{8}$	$4\frac{1}{4}$
5	$4\frac{7}{8}$	$5\frac{1}{4}$
6	$5\frac{3}{4}$	$6\frac{1}{4}$
8	$7\frac{3}{4}$	$8\frac{1}{4}$

(*c*) *Dimension Determinations.*—Overall dimensions shall be measured from opposite face to opposite face of the tile or block, the greatest measurement observed being taken as the dimension.

Sectional dimensions of cored tile shall be taken 1 in. in from the ends of the tile or block, the least measurement observed being taken as the dimension.

Shell dimensions shall be measured from the surface of the core space to the normal surface of the tile or block inside the scoring.

(*d*) *Dimension Tolerances.*—A variation of $\frac{1}{4}$ in. in the nominal width and $\frac{3}{8}$ in. in the nominal length of the tile or block is permissible.

(*e*) *Scoring.*—The scoring upon gypsum tile or block shall be of such a nature as not to materially reduce the shell thickness.

PHYSICAL PROPERTIES AND TESTS

Strength.

7. (*a*) When tested in accordance with the methods described in Section 14, the compressive strength of gypsum tile or block shall not be less than 75 lb. per sq. in. for the average of five specimens tested.

(*b*) When completely saturated, the compressive strength shall not be less than 33⅓ per cent of the strength required for such tile or block when dried to constant weight.

(*c*) When tested in accordance with Section 16 the rate of absorption of gypsum tile or block shall not be less than 8 cc. nor more than 30 cc. for the first minute, nor less than 4 cc. for each of the next five subsequent minutes. **Absorption.**

8. In locations where fire resistance is an essential property, gypsum tile or block shall meet the requirements of the Tentative Specifications for Fire Tests of Building Construction and Materials (Serial Designation: C 19 – 26 T) of the American Society for Testing Materials,[1] as they apply for the construction in which the tile are used. **Fire Resistance.**

SAMPLING

9. At least 1 per cent of the number of gypsum tile or block in a shipment, but in no case less than ten tile, shall be so selected as to be representative of the shipment. Twenty-five per cent of such selected tile, but in no case less than ten individual tile, shall constitute a sample for test purposes. In case a shipment consists of more than one car or carrier load, a sample shall be selected from each car or carrier. Samples for test shall be selected at the factory or at the point of delivery, as specified in the sales contract. **Sampling.**

MARKING

10. When gypsum tile or block are shipped for resale, the manufacturer's "Brand" name shall be shown upon each tile. **Marking.**

INSPECTION AND REJECTION

11. Inspection may be made either at the point of shipment or at the point of delivery. The inspector representing the purchaser shall have free access to the carriers being loaded for shipment to the purchaser. He shall be afforded all reasonable facilities for inspection and sampling, which shall be so conducted as not to interfere unnecessarily with the loading of the carriers. **Inspection.**

12. Any rejection shall be based upon failure to conform to the requirements of these specifications, and shall be reported within ten working days from the receipt of the shipment by the consignee. The notice of rejection shall contain a specific statement of the respects in which the tile or block have failed to meet the requirements of these specifications. **Rejection.**

13. Claims for rehearing shall be valid only if made within twenty working days from receipt of notice of specific cause for rejection. **Rehearing.**

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I, p. 761 (1926); also 1927 Book of A.S.T.M. Tentative Standards, p. 291.

METHODS OF TESTING

COMPRESSIVE STRENGTH OF GYPSUM PARTITION TILE OR BLOCK

Compressive Strength of Tile or Block.

14. (*a*) Not less than ten full-size gypsum tile specimens shall be used.

(*b*) The test specimens shall be dried to constant weight at a temperature not exceeding 110° F. (43° C.), until two successive weighings of the same specimen do not show a variation in excess of 0.5 per cent. At the option of the manufacturer or purchaser, strength tests may be conducted on samples that are dried at room temperature. However, in cases of controversy all test samples shall be dried to constant weight as herein prescribed.

(*c*) The tile or block shall be tested in the position in which they are designed to be used and shall be bedded on and capped with a felt gasket not less than $\frac{1}{8}$ in. nor more than $\frac{1}{4}$ in. in thickness. At the option of the manufacturer or purchaser, or in cases of controversy, the test samples may be suitably bedded and capped with neat gypsum mortar, or the bearing surfaces of the tile may be planed or rubbed smooth and true. When neat gypsum mortar is used for bedding and capping, the test may be conducted after the mortar has set, but not sooner than twenty-four hours after the sample has been capped.

(*d*) The loading head shall completely cover the bearing area of the tile, and the applied load shall be transmitted through a spherical bearing block of proper design. The speed of the moving head of the testing machine shall not be more than 0.05 in. per minute.

TRANSVERSE STRENGTH OF GYPSUM PARTITION TILE OR BLOCK

Transverse Strength of Tile or Block.

15. (*a*) Not less than ten full-size gypsum tile specimens shall be used.

(*b*) The test specimens shall be dried to constant weight as prescribed for the determination of the compressive strength in accordance with Section 14(*b*).

(*c*) The tile or block shall be supported on its face (flat) on fixed parallel bearings spaced 24 in. (600 mm.) on centers, and shall be loaded through a similar bearing midway between the supports. All bearing and load surfaces shall be true, shall engage the full width of the test specimen, and shall be rounded to a radius of $\frac{1}{2}$ in. (12.7 mm.).

(*d*) The speed of the moving head of the testing machine shall not be more than 0.05 in. per minute. The modulus of rupture shall be calculated in pounds per square inch from the formula:

$$\text{Modulus of Rupture} = \frac{3\,wl}{2bd^2}$$

where l = distance between supports in inches;
b = breadth (width) of tile in inches;
d = thickness of tile in inches;
w = load in pounds at failure of test specimen.

ABSORPTION OF GYPSUM PARTITION TILE OR BLOCK

Absorption of Tile or Block.

16. (*a*) The absorption shall be determined upon a test specimen of partition tile or block consisting of approximately one-half of a full-size tile.

(*b*) The test specimen shall be dried to constant weight as prescribed for the determination of the compressive strength in accordance with Section 14(*b*).

(*c*) The dried test specimen shall be submerged in water until a constant saturated weight is attained, but in no event for a period of less than two hours, the water temperature being maintained at between 70 and 80° F. (21 and 27° C.). The test specimen shall be removed, the surface wiped off with a damp cloth and the specimen weighed immediately. The percentage of absorption shall be calculated on the dry weight from the formula:

$$\text{Percentage of Absorption} = \frac{100\,(B-A)}{A}$$

where A = weight of dry test specimen;
B = weight of saturated test specimen.

(*d*) The rate of absorption shall be determined by sealing upon the face of the test specimen, which shall first be dried to constant weight as prescribed for determination of the compressive strength, a glass tube $1\frac{1}{2}$ in. internal diameter and 12 in. in length, graduated in centimeters and located centrally over a core. Into this tube shall be poured 250 cc. of water at a temperature of between 70 and 80° F. (21 and 27° C.). The rate of absorption shall be reported in cubic centimeters per minute.

STANDARD METHODS OF TESTING GYPSUM AND GYPSUM PRODUCTS[1]

Serial Designation: C 26 – 27

These methods are issued under the fixed designation C 26; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1919; ADOPTED IN AMENDED FORM, 1923; REVISED, 1927.

DETERMINATION OF FREE WATER IN GYPSUM

Free Water.

1. (*a*) Not less than 1 lb. of the entire sample as received shall be weighed, spread out in a thin layer in a suitable vessel, placed in a drying oven, and dried at 45° C. for 2 hours. It shall then be cooled in an atmosphere free from moisture, and weighed again. The loss of weight corresponds to the free water, and shall be calculated to percentage of sample as received.

(*b*) The dried sample shall be stored in an air-tight container until used.

DETERMINATION OF FINENESS

Fineness.

2. Fineness shall be determined by sieving a known weight of the dried sample through sieves of the specified sizes and weighing the amount of material retained on each sieve. The material shall be shaken through the sieve with as little abrasion as possible.

The size of the sample to be used in this determination shall depend upon the size of grain of the sample. If the sample will all pass a $\frac{1}{4}$-in. sieve, 100 g. will be sufficient; if the largest particles are more than 1 in. in diameter, at least 1000 g. must be used. With these limitations the size of sample to be used is left to the discretion of the operator.

NOTE.—The sizes of the sieves to be used are given in the Standard Specifications for Gypsum (Serial Designation: C 22),[2] for Calcined Gypsum (Serial Designation: C 23),[3] and for Gypsum Plasters (Serial Designation: C 28)[4] of the American Society for Testing Materials, covering the particular product examined.

CHEMICAL ANALYSIS OF GYPSUM AND CALCINED GYPSUM

Preparation of Sample.

3. Dry the sample as described in Section 1, and reduce about 10 g. until it all passes a No. 100 (149-micron) sieve,[5] using extreme care

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

[2] See p. 72. [3] See p. 75. [4] See p. 78.

[5] For detailed specifications for these sieves see the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

not to unduly expose the material to the action of moisture or to overheating. The sample shall be kept in an air-tight container until ready for use.

Combined Water.

4. Place 1 g. of the sample in a covered crucible and dry at 215 to 230° C. to constant weight. Calculate the loss of weight to percentage of sample as received and report as combined water.

Carbon Dioxide.

5. Place the residue obtained after drying, as described in Section 4, in a suitable flask and dissolve it in dilute HCl (not stronger than 1:4) in such a way that the gas evolved, after being freed from H_2O vapor by calcium chloride or sulfuric acid, can be collected in either soda-lime or caustic potash and weighed. The solution should be boiled for one minute, and a current of CO_2-free air kept passing through the apparatus for 30 minutes. The increase of weight of the soda-lime or caustic potash corresponds to the weight of carbon dioxide, which is to be calculated to percentage of sample as received.

Silica and Insoluble.

6. Place 0.5 g. of the sample prepared as described in Section 3 in a porcelain casserole. Add about 25 cc. of HCl (1:5), and evaporate to apparent dryness on a hot plate. Cool and add enough concentrated HCl to wet thoroughly. Add about 10 cc. of water, boil, filter, and wash. Put the filtrate back in the same casserole. Evaporate it to dryness and heat to about 120° C. for one hour. Cool. Add enough concentrated HCl to wet thoroughly. Add about 25 cc. of water, boil, filter, and wash. Transfer the two papers containing the two precipitates to the same crucible, ignite, and weigh. Calculate this weight to percentage of sample as received.

Iron and Alumina.

7. To the filtrate obtained as described in Section 6, add a few drops of HNO_3 and boil to insure oxidation of the iron. Add 2 g. of NH_4Cl previously dissolved in water. Make alkaline with NH_4OH. Digest hot for a few minutes until the precipitate coagulates. Filter, wash, ignite the precipitate and weigh as $Fe_2O_3 + Al_2O_3$. Calculate this weight to percentage of sample as received. This precipitate may be further treated to separate the two oxides, but this is generally unnecessary.

Lime.

8. (*a*) To the filtrate obtained as described in Section 7, add 5 g. of $(NH_4)_2C_2O_4$ dissolved in water. Digest hot for ½ hour, making sure that the solution is always alkaline with NH_4OH. Filter, wash and ignite in a platinum crucible over a strong blast to constant weight. Calculate this weight to percentage of sample as received.

(*b*) *Alternative Method.*—To the filtrate obtained as described in Section 7, add 5 g. of $(NH_4)_2C_2O_4$ dissolved in water. Digest hot for ½ hour, making sure that the solution is always alkaline with NH_4OH. Filter and wash. Transfer the precipitate to a beaker, and wash the filter paper with hot dilute H_2SO_4, catching the wash-

ings in the same beaker. Heat gently to complete solution, adding more H_2SO_4 if necessary. While still warm, titrate with a solution of $KMnO_4$ containing 5.6339 g. per liter, until the pink color is permanent. The number of cubic centimeters of $KMnO_4$ used gives directly the percentage of lime in the dried sample. Recalculate to percentage of sample as received.

Magnesia. 9. To the filtrate obtained as described in Section 8 (*a*) or (*b*), add enough water to give a total volume of about 600 cc. Cool. Add 10 cc. of NH_4OH and 5 g. $NaNH_4HPO_4$ dissolved in water. Stir until precipitate begins to form. Let stand over night. Filter, wash with a 2.5-per-cent by weight solution of NH_4NO_3. Ignite and weigh. Multiply this weight by 40/111 to find the weight of MgO. Then calculate to percentage of sample as received.

Sulfur Trioxide. 10. Dissolve 0.5 g. of the sample prepared as described in Section 3 in 50 cc. of HCl (1:5). Boil. Add 100 cc. of boiling water, and continue boiling for 5 minutes. Filter immediately and wash thoroughly with hot water. Boil, and while boiling, add slowly 20 cc. of a boiling solution of $BaCl_2$ (10-per-cent). Digest hot for one hour, or until precipitate settles. Filter and wash. Dry carefully. Ignite over Bunsen burner at lowest heat possible until filter paper is burned off. Ignite at bright red heat for 15 minutes, and weigh. Multiply this weight by 80/233 to determine the weight of SO_3. Then calculate to percentage of sample as received.

Sodium Chloride. 11. Dissolve in boiling water a 1-g. sample (prepared as described in Section 3), put on the filter and wash with 250 cc. of boiling water, then titrate the filtrate. Add two or three drops of potassium chromate solution and titrate with an 0.05 *N* solution of silver nitrate. Each cubic centimeter of silver nitrate solution equals 0.002923 g. of sodium chloride. Calculate to percentage of sample tested.

Calculation of Results. 12. By the methods given above, the results are obtained and reported in the following form:

	PER CENT.
Free water	
Combined water	
Silica and insoluble, SiO_2	
Iron and alumina, R_2O_3	
Lime CaO	
Magnesia, MgO	
Sulfur Trioxide, SO_3	
Carbon Dioxide, CO_2	
Sodium Chloride, NaCl	
	100.00

NOTE

Since it is frequently advisable to recalculate these results, that they may be more enlightening, the following method is submitted for consideration:

(*a*) Multiply percentage of MgO by 84/40, to find percentage of $MgCO_3$.

(*b*) Multiply the percentage of MgO by 44/40 to find the percentage of CO_2 as $MgCO_3$.

(*c*) Deduct CO_2 as $MgCO_3$ from the CO_2 determined.

(*d*) Multiply the CO_2 remaining by 100/44 to find percentage of $CaCO_3$.

(*e*) Add together the percentage of SiO_2 R_2O_3, $MgCO_3$, and $CaCO_3$, and report in the aggregate.

(*f*) Multiply the percentage of $CaCO_3$ by 56/100 to find the percentage of CaO as $CaCO_3$.

(*g*) From the total percentage of CaO, deduct the percentage of CaO as $CaCO_3$. The remainder may be called "available CaO."

(*h*) The "available CaO" should bear to the SO_3 a ratio of 7 to 10. Determine which (if either) is in excess.

(*i*) If the CaO is in excess, multiply the SO_3 by 7/10, and subtract the result from the "available CaO." The remainder is reported as "excess CaO."

(*j*) If the SO_3 is in excess, multiply the "available CaO" by 10/7 and subtract the result from the SO_3. The remainder is reported as "excess SO_3."

(*k*) Add together the "available CaO," and the SO_3, and subtract the "excess CaO" or "excess SO_3." The remainder is $CaSO_4$.

(*l*) If the $CaSO_4$ is present as $CaSO_4 \cdot \frac{1}{2}H_2O$, the percentage of $CaSO_4$ should bear to the percentage of combined water a ratio of 136 to 9. Determine which (if either) is in excess.

(*m*) If the $CaSO_4$ is in excess some of it is present in the anhydrous form. Multiply the percentage of combined water by 136/9 to find the percentage of $CaSO_4$ as $CaSO_4 \cdot \frac{1}{2}H_2O$. The difference between the total $CaSO_4$ and the percentage of $CaSO_4$ as $CaSO_4 \cdot \frac{1}{2}H_2O$, is the $CaSO_4$ in the anhydrous form.

(*n*) If the water is in excess, some of the $CaSO_4$ is present as gypsum. Let x = percentage of $CaSO_4 \cdot \frac{1}{2}H_2O$, and y = percentage of $CaSO_4 \cdot 2H_2O$. Then $x + y$ = percentage of $CaSO_4$ (as found in *k*) + percentage of water.

$$\frac{9x}{145} + \frac{36y}{172} = \text{percentage of combined water.}$$

Solve these equations for x and y. Report x as percentage of "calcined gypsum," $CaSO_4 \cdot \frac{1}{2}H_2O$. Report y as percentage of gypsum, $CaSO_4 \cdot 2H_2O$.

Having made these calculations, the result may be reported as follows:

	PER CENT.
Gypsum, $CaSO_4 + 2H_2O$	
Calcined gypsum, $CaSO_4 + \frac{1}{2}H_2O$	
Anhydrite, $CaSO_4$	
Excess CaO or Excess SO_3	
Sodium chloride, NaCl	
Other ingredients	
	100.00 ±

The presence of the different forms of $CaSO_4$ may be corroborated by a microscopic examination.

PRECAUTIONS FOR PHYSICAL TESTS

Precautions for Physical Tests.

13. Gypsum products are peculiar in that their properties are very greatly affected by the small amounts of impurities which may be introduced by careless laboratory manipulation. In order to obtain concordant results, it is, therefore, absolutely essential to observe the following precautions:

(*a*) All apparatus shall be kept thoroughly clean. Especially shall all traces of set plaster be removed.

(*b*) Distilled water, free from chlorides and sulfates, shall be used throughout.

(*c*) Standard Ottawa sand shall be used throughout. This shall be prepared for use by washing it once with HCl (1:4) and four times with distilled water, and then drying it.

TESTING CONSISTENCY OF CALCINED GYPSUM

Testing Consistency.

14. In order that the results of testing samples of different plaster shall be directly comparable, it is necessary that all calcined gypsum be first brought to the same consistency, by the addition of the proper amount of water. An accurate method for determining this consistency is the most important step in the standardizing of physical methods for testing cementing materials.

Apparatus.

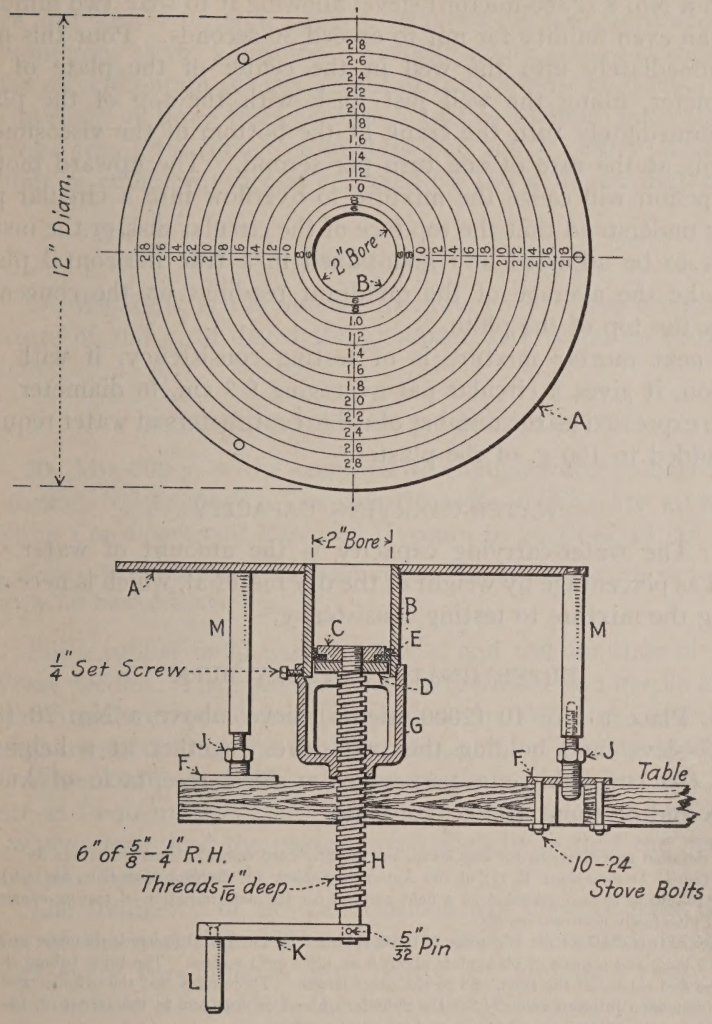
15. For a description of the apparatus to be used for measuring consistency, reference is made to the accompanying illustration (Fig. 1) of the Southard viscosimeter which has been used with satisfactory results.

The apparatus consists of a brass cylinder of 2-in. (5.08 cm.) bore with a circular disk flange flush with its upper end. The screw actuating the piston is $\frac{5}{8}$ in. (1.59 cm.) in outside diameter, $\frac{1}{4}$-in. (0.635-cm.) pitch, right-hand square threads $\frac{1}{16}$ in. (0.159 cm.) deep. The top of the brass disk flange is etched with concentric circles which vary in diameter from 6 cm. up to 28 cm. by increments of 2 cm.

When in position for use the brass flange is to be maintained in a true horizontal position.

Method.

16. To prepare this viscosimeter for use see that the piston, cylinder walls and top of plate are clean. Then by turning the crank, bring the top of the piston exactly flush with the top of the plate. Then by reverse cranking make ten complete turns, which will lower the piston to a point $2\frac{1}{2}$ in. (6.35 cm.) below the top of the plate. Make a mixture of at least 300 g. total of dry calcined gypsum and water. Shake the calcined gypsum into the water



A; 3/16" Hard Brass Plate Scale Disk.
B; 2" Ex. H. Seamless Brass Tube Cylinder.
C; Brass Piston Top.
D; " " Bottom.
E; Leather " Packing.
F; 3/16" x 1" Flat Bar Steel Support Plate.
G; Brass Cage (Open on two Sides)
H; 5/8" Hard Brass Piston Screw.
J; 1/2" Hex. Brass Support Stud.
K; 3/8" x 3/4" C. R. S. Screw Lever.
L; 3/8" Diam. C. R. S. Lever Handle.
M; 1/2" Diam. C. R. S. Support Rod.

FIG. 1.—Southard Viscosimeter.

through a No. 8 (2380-micron) sieve[1] allowing it to soak two minutes. Stir to an even fluidity for not to exceed 30 seconds. Pour this mixture immediately into the well in the center of the plate of the viscosimeter, filling the well just flush with the top of the plate. Then immediately turn the crank at the bottom of the viscosimeter ten turns at the rate of one turn per second. The upward motion of the piston will cause the mixture to overflow into a circular pat, it being understood that the top face of the circular disk of the instrument is to be adjusted and maintained in a true horizontal plane. Next take the average of the quadrant readings on the concentric lines on the top of the plate.

A neat mortar mixture is of testing consistency, if with this operation, it gives a circular pat averaging 9.7 cm. in diameter, and shall be expressed as the number of cubic centimeters of water required to be added to 100 g. of the plaster.[2]

WATER-CARRYING CAPACITY

Water-Carrying Capacity.

17. The water-carrying capacity is the amount of water, calculated as percentage by weight of the dry material, which is necessary to bring the mixture to testing consistency.

DETERMINATION OF DRY BULK

Dry Bulk.

18. Place a No. 10 (2000-micron) sieve[1] above a No. 20 (840-micron) sieve[1] and holding the two sieves together at a height of 12 in. (30 cm.) above a perpendicular-sided receptacle of known volume and of about 400 cc. content.

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[1] For detailed specifications for this sieve, see the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

[2] The following is recommended as a field method for the determination of testing consistency which will give fairly accurate results:

The apparatus shall consist of a piece of brass tubing 2 in. (5.08 cm.) in inside diameter and 4 in. (10.15 cm.) long, and a piece of plate glass about 8 in. (20.3 cm.) square. The brass tubing shall be cut off true and square at the ends, and to the exact length. Thoroughly wet the cylinder and glass plate by immersion in clean water. Set the cylinder on end on top, and in the center of, the glass plate which is set and maintained in a true horizontal position.

Prepare a dry sample of calcined gypsum. Next make a mixture of calcined gypsum and water of sufficient quantity to slightly more than fill the cylinder. The mixture shall be made by adding the calcined gypsum to the water, allowing it to soak for two minutes. Now stir to an even fluidity for not to exceed 30 seconds, and at once pour this mixture into the cylinder so as to completely fill it level with the top.

Immediately raise the cylinder from the glass with a quick, straight and upward motion so as to withdraw it from the mixture within. This act will allow the mixture to leave the cylinder, assuming a cone-shaped circular pat upon the glass plate.

The testing consistency of a neat mortar is a mixture which will give a circular pat averaging 4¾ in. (12 cm.) in diameter, and shall be expressed as the number of cubic centimeters of water required to be added to 100 g. of the plaster.

The testing consistency of a sanded mortar is a mixture that, when placed in a 2 by 4-in. (5.08 by 10.15-cm.) cylinder immediately removed, the slump or settlement of the sanded mixture will be ½ in. (1.27 cm.).

Place the material under test on top of the No. 10 (2000-micron) sieve and pass it through the No. 10 (2000-micron) sieve and No. 20 (840-micron) sieve, filling the receptacle under the 12-in. head, then strike off and obtain the net weight of the contents in grams. Obtain the average of three tests according to this method and express the average result in cubic centimeters per 100 g. Dry Bulk is the volume in cubic centimeters occupied by 100 g. of dry material.[1]

DETERMINATION OF WET BULK

Wet Bulk.

19. Wet bulk is the volume in cubic centimeters occupied by a mixture of 100 g. of dry material mixed with water to testing consistency.

DETERMINATION OF TIME OF SETTING

Time of Setting.

20. Mix 200 g. of the sample with enough water to make a paste of testing consistency. The directions for mixing are given under Testing Consistency of Calcined Gypsum in Sections 14, 15 and 16.

NOTE.—If the sample is a sanded mixture use 300 g. and mix to testing consistency for sanded mixture.

Fill a rubber mold with the paste, and test for time of set with a Vicat needle. The specifications for the mold and needle are given in the Standard Specifications and Tests for Portland Cement (Serial Designation: C 9) of the American Society for Testing Materials.[2]

When conducting the test, the needle is allowed to sink into the paste at frequent intervals. After each penetration, the needle must be wiped clean, and the paste moved slightly so that the needle will not strike the same hole twice.

The frequency of the penetrations will depend upon the character of the material. If the sample is not retarded, it should be tested every two minutes until nearly set, and then every minute. If the sample is retarded, the intervals at first may be as long as one hour, but in any case they must be so selected that the time of set is ascertained to the nearest minute.

Set is considered complete when the needle no longer penetrates to the bottom of the paste. The minutes elapsed, from the time when the sample was first added to the water, to the time when set is complete, is recorded as the time of set of the sample.

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[1] Dry Bulk is expressed as the volume in cubic centimeters occupied by 100 g. of dry material instead of "grams per 100 cc.," since Bulk is understood to mean Volume per Unit Weight rather than Weight per Unit Volume.

[2] See p. 23.

Caution: For test to determine the time of set, it is absolutely essential that all dishes and utensils be clean. Especially must they be free from all traces of set gypsum. Use distilled water.

DETERMINATION OF TENSILE STRENGTH

Tensile Strength.

21. Mix 500 g. of the sample to testing consistency. Cast into a five-gang briquet mold, of the shape and size used for testing portland cement, see the Standard Specifications and Tests for Portland Cement (Serial Designation: C 9) of the American Society for Testing Materials.[1] Do not cast each briquet successively, but move the containing vessel back and forth over the molds while pouring continuously. Work the briquet slightly with the point of the trowel to remove air bubbles, and level off the briquets. When sufficiently hard, remove and store in the room at a temperature of not less than 15.6° C. (60° F.) nor more than 37.8° C. (100° F.) for at least seven days. Weigh once a day. When the weight has become constant to within 0.1 per cent, proceed to test the specimens in a standard machine used for the determination of tensile strength.

NOTE.—When the material to be tested is a sanded mixture, use not less than 750 g. of the sample and mix to testing consistency for sanded mixture.

The average tensile strength shall be reported as the tensile strength of the material, except that if one or two briquets vary more than 15 per cent from the average of the five, they shall be discarded and the tensile strength shall be reported as the average of the remaining specimens. In case three or more briquets vary more than 15 per cent from the average of the five, tests shall be continued upon three batches of five briquets and the average of the fifteen briquets shall be reported. The tensile strength of all briquets shall be reported.

DETERMINATION OF COMPRESSIVE STRENGTH

Compressive Strength.

22. Mix 2000 g. of the sample to testing consistency. Cast into five 2 by 4-in. split cylinders set plumb upon a metal or glass plate. Do not cast each cylinder successively, but move the containing vessel back and forth over the cylinders while pouring continuously. Work the cylinders slightly with the point of the trowel to remove air bubbles, and level off the top of the cylinders. When sufficiently hard, remove and store in the room at a temperature of not less than 15.6° C. (60° F.) nor more than 37.8° C. (100° F.) for at least seven days, and by weighing once a day, when the weight has become constant to within 0.1 per cent, proceed to test the speci-

[1] See p. 23.

mens in a standard machine used for the determination of compressive strength.

NOTE.—When the material to be tested is a sanded mixture use not less than 3000 g. of the sample and mix to testing consistency for sanded mixture.

The average compressive strength shall be reported as the compressive strength of the material, except that if one or two of the cylinder specimens vary more than 15 per cent from the average, the compressive strength shall be reported as the average of the remaining specimens. The compressive strength of all the cylinders shall be reported.

COMPRESSIVE STRENGTH OF GYPSUM PARTITION TILE OR BLOCK

Compressive Strength of Tile or Block.

23. (*a*) Not less than ten full-size gypsum tile specimens shall be used.

(*b*) The test specimens shall be dried to constant weight at a temperature not exceeding 110° F. (43° C.), until two successive weighings of the same specimen do not show a variation in excess of 0.5 per cent. At the option of the manufacturer or purchaser, strength tests may be conducted on samples that are dried at room temperature. However, in cases of controversy all test samples shall be dried to constant weight as herein prescribed.

(*c*) The tile or block shall be tested in the position in which they are designed to be used and shall be bedded on and capped with a felt gasket not less than $\frac{1}{8}$ in. nor more than $\frac{1}{4}$ in. in thickness. At the option of the manufacturer or purchaser, or in cases of controversy, the test samples may be suitably bedded and capped with neat gypsum mortar, or the bearing surfaces of the tile may be planed or rubbed smooth and true. When neat gypsum mortar is used for bedding and capping, the test may be conducted after the mortar has set, but not sooner than twenty-four hours after the sample has been capped.

(*d*) The loading head shall completely cover the bearing area of the tile, and the applied load shall be transmitted through a spherical bearing block of proper design. The speed of the moving head of the testing machine shall not be more than 0.05 in. per minute.

TRANSVERSE STRENGTH OF GYPSUM PARTITION TILE OR BLOCK

Transverse Strength of Tile or Block.

24. (*a*) Not less than ten full-size gypsum tile specimens shall be used.

(*b*) The test specimens shall be dried to constant weight as prescribed for the determination of the compressive strength in accordance with Section 23 (*b*).

(*c*) The tile or block shall be supported on its face (flat) on fixed parallel bearings spaced 24 in. (600 mm.) on centers, and shall be loaded through a similar bearing midway between the supports. All bearing and load surfaces shall be true, shall engage the full width of the test specimen, and shall be rounded to a radius of $\frac{1}{2}$ in. (12.7 mm.).

(*d*) The speed of the moving head of the testing machine shall not be more than 0.05 in. per minute. The modulus of rupture shall be calculated in pounds per square inch from the formula:

$$\text{Modulus of Rupture} = \frac{3wl}{2bd^2}$$

where l = distance between supports in inches;
b = breadth (width) of tile in inches;
d = thickness of tile in inches;
w = load in pounds at failure of test specimen.

ABSORPTION OF GYPSUM PARTITION TILE OR BLOCK

Absorption of Tile or Block.

25. (*a*) The absorption shall be determined upon a test specimen of partition tile or block consisting of approximately one-half of a full-size tile.

(*b*) The test specimen shall be dried to constant weight as prescribed for the determination of the compressive strength in accordance with Section 23 (*b*).

(*c*) The dried test specimen shall be submerged in water until a constant saturated weight is attained, but in no event for a period of less than two hours, the water temperature being maintained at between 70 and 80° F. (21 and 27° C.). The test specimen shall be removed, the surface wiped off with a damp cloth and the specimen weighed immediately. The percentage of absorption shall be calculated on the dry weight from the formula:

$$\text{Percentage of Absorption} = \frac{100\ (B - A)}{A}$$

where A = weight of dry test specimen;
B = weight of saturated test specimen.

(*d*) The rate of absorption shall be determined by sealing upon the face of the test specimen, which shall first be dried to constant weight as prescribed for determination of the compressive strength, a glass tube $1\frac{1}{2}$-in. internal diameter and 12 in. in length, graduated in centimeters and located centrally over a core. Into this tube shall be poured 250 cc. of water at a temperature of between 70 and 80° F. (21 and 27° C.). The rate of absorption shall be reported in cubic centimeters per minute.

TRANSVERSE STRENGTH OF GYPSUM BOARDS

Transverse Strength of Gypsum Boards.

26. (*a*) Test specimens shall be taken from not less than ten gypsum boards.

(*b*) Strength test specimens shall be 12 in. (300 mm.) in width and approximately 18 in. (460 mm.) in length. They shall be supported on fixed parallel bearings spaced 16 in. (410 mm.) on centers, and shall be loaded through a similar bearing midway between the supports. All bearing and load surfaces shall be true, shall engage the full width of the test specimen, and shall be rounded to a radius of $\frac{1}{8}$ in. (3.2 mm.).

(*c*) Test loads shall be applied at a uniform rate of 60 lb. (27.22 kg.) per minute with a permissible variation of ± 10 per cent in the rate. Strength test results shall be reported when the load is applied across the fiber of the surfacing and also parallel to the fiber of the surfacing.

STANDARD DEFINITIONS OF TERMS RELATING TO THE GYPSUM INDUSTRY[1]

Serial Designation: C 11 – 26

These definitions are issued under the fixed designation C 11; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1916; ADOPTED IN AMENDED FORM, 1926.

Accelerator.—In the case of stucco, plaster, mortar, concrete, etc., a substance which will hasten the set.

Binder.—In the case of plaster and stucco, etc., a fibrous material, which will increase their cohesiveness, while they are in a plastic state.

Cement.—A material or a mixture of materials (without aggregate) which, when in a plastic state, possesses adhesive and cohesive properties, and which will harden in place.

NOTE.—The word "cement" is used without regard to the composition of the material.

Calcined Gypsum.—Gypsum partially dehydrated by means of heat, having the approximate chemical formula $CaSO_4 \cdot \frac{1}{2} H_2O$.

Crushed Gypsum.—Gypsum subjected to a primary crushing operation.

Gypsum.—Calcium sulfate, combined with two molecules of water, in crystallin form, having the approximate chemical formula $CaSO_4 \cdot 2H_2O$.

Gypsum Plastering Sand.—The fine granular material naturally or artificially produced by the disintegration of rock, containing not less than 80 per cent by weight of silica, feldspar, dolomite, magnesite or calcite.

Lath.—A material whose primary function is that of a base or background for the reception of plaster or stucco.

Mortar.—A material used in a plastic state, which can be troweled, and becomes hard in place, to bond units of masonry structures.

NOTE.—The word "mortar" is used without regard to the composition of the material, and is defined only with reference to its use as a bonding material as contrasted with the words "stucco" and "plaster."

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

Plaster.—A material used in a plastic state, which can be troweled, to form a hard covering for the interior surfaces, walls and ceilings, etc., in any building or structure.

NOTE 1.—The word "plaster" is used without regard to the composition of the material, and is defined only with reference to its use and location of use as contrasted with the words "stucco" and "mortar."

NOTE 2.—This does not include decorative media ordinarily applied with a brush.

Retarder.—In the case of stucco, plaster, mortar, concrete, etc., a substance which will retard the set.

Sized Gypsum.—Crushed gypsum of prescribed size of individual particles.

Stucco.—A material used in a plastic state, which can be troweled, to form a hard covering for the exterior walls or other exterior surfaces of any building or structure.

NOTE.—The word "stucco" is used without regard to the composition of the material, and is defined only with reference to its use and location of use as contrasted with the words "plaster" and "mortar."

Wood Fiber.—A material produced by grinding or shredding wood.

STANDARD METHOD

OF

MAKING AND STORING SPECIMENS OF CONCRETE IN THE FIELD[1]

Serial Designation: C 31 – 27

This method is issued under the fixed designation C 31; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1920; ADOPTED, 1921; REVISED, 1927.

Scope.

1. This method is intended to apply to the molding and storing of test specimens of concrete sampled from concrete being used in construction.

Size and Shape of Specimens.

2. The test specimens shall be cylindrical in form with the length twice the diameter. The standard shall be 6 by 12-in. cylinders where the coarse aggregate does not exceed 2 in. in size; for aggregates larger than 2 in., 8 by 16-in. cylinders shall be used; 2 by 4-in. cylinders may be used for mixtures without coarse aggregate.

Molds.

3. (*a*) The molds shall be cylindrical in form, made of non-absorbent material, and shall be substantial enough to hold their form during the molding of the test specimens. They shall not vary in diameter more than $\frac{1}{16}$ in. in any direction, nor shall they vary in height more than $\frac{1}{16}$ in. from the height required. They shall be substantially water tight so that there will be no leakage of water from the test specimen during molding.

NOTE.—The best type of mold consists of lengths of cold-drawn steel tubing, split along one element and closed by means of a circumferential band and bolt. Satisfactory molds can be made from lengths of steel water pipe machined on the inside, from rolled metal plates, from galvanized steel, machined iron or steel castings. Paraffined cardboard molds will give good results under expert supervision.

(*b*) Each mold shall be provided with a base plate having a plane surface and made of non-absorbent material. This plate shall be large enough in diameter to properly support the mold without leakage. Plate glass or planed metal are satisfactory for this purpose.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

A similar plate should be provided for covering the top surface of the test specimen when molded.

(*c*) Suggestions for suitable molds are shown in Figs. 1, 2, and 3.

4. (*a*) Concrete for the test specimens shall be taken immediately after it has been placed in the work. All the concrete for each sample shall be taken from one place. A sufficient number of samples—each large enough to make one test specimen—shall be taken at

Sampling of Concrete.

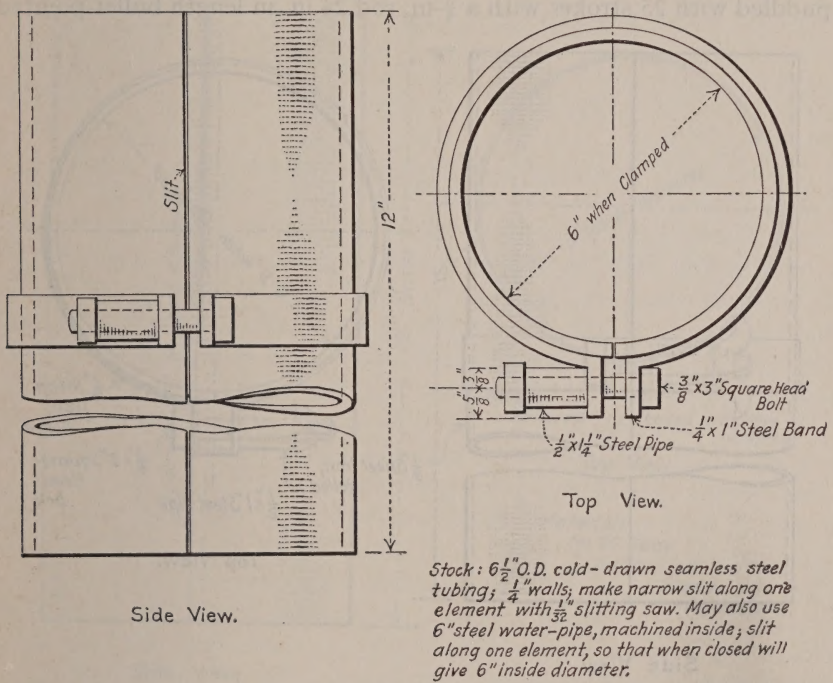
Slit
12"
6" when Clamped
3/8" x 3" Square Head Bolt
1" x 1" Steel Band
1/2" x 1 1/4" Steel Pipe
Top View.
Side View.
Stock: 6 1/2" O.D. cold-drawn seamless steel tubing; 1/4" walls; make narrow slit along one element with 1/32" slitting saw. May also use 6" steel water-pipe, machined inside; slit along one element, so that when closed will give 6" inside diameter.

FIG. 1.

different points so that the test specimens made from them will give a fair average of the concrete placed in that portion of the structure selected for tests. The location from which each sample is taken shall be noted clearly for future reference.

(*b*) In securing samples, the concrete shall be taken from the mass by a shovel or similar implement and placed in a large pail or other receptacle, for transporting to the place of molding. Care shall be taken to see that each test specimen represents the total mixture of the concrete from a given place. Different samples shall not be mixed together but each sample shall make one specimen.

Molding Test Specimens.

5. (*a*) The pails or other receptacles containing the samples of concrete shall be taken as quickly as possible to the place selected for molding test specimens. To offset segregation of the concrete occurring during transportation, each sample shall be dumped into a non-absorbent water-tight receptacle and, after slight mixing, immediately placed in the mold.

(*b*) The test specimens shall be molded by placing the concrete in the mold in layers 3 to 4 in. in thickness. Each layer shall be puddled with 25 strokes with a $\frac{5}{8}$-in. rod 24 in. in length bullet-pointed

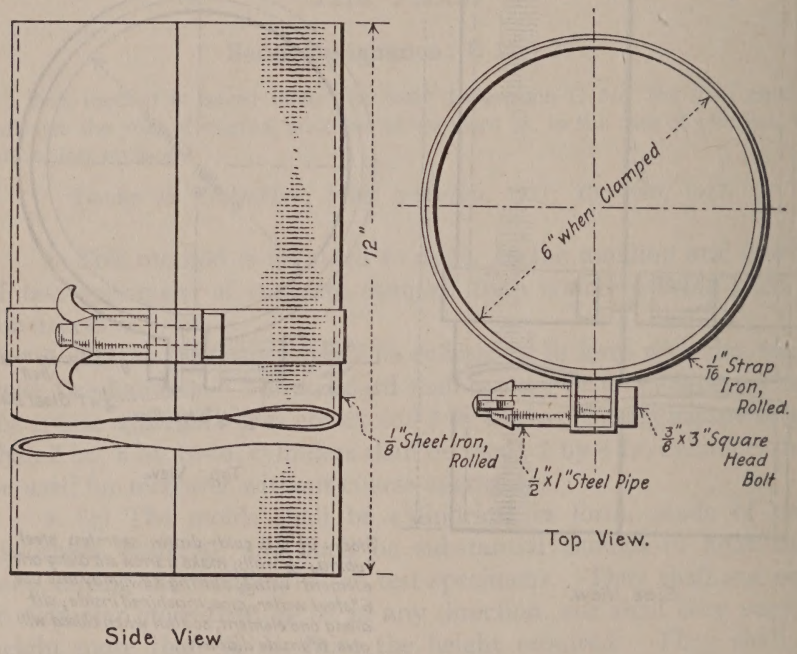

FIG. 2.

at the lower end. After puddling the top layer, the surface concrete shall be struck off with a trowel and covered with the top cover plate which may later be used in capping the test specimen.

Capping Specimens.

6. Two to four hours after molding, the test specimens shall be capped with a thin layer of stiff neat-cement paste in order that the cylinder may present a smooth end for testing. The cap shall be formed by means of a piece of plate glass $\frac{1}{4}$ in. in thickness or a machined metal plate $\frac{1}{2}$ in. in thickness and of a diameter 2 or 3 in. larger than that of the mold. The plate shall be worked on the fresh cement paste until it rests on top of the mold. The cement for capping should be mixed to a stiff paste 2 to 4 hours before it is to be used in

order to avoid the tendency of the cap to shrink. Adhesion of the concrete to the top and bottom plates may be avoided by oiling the plates or by inserting a sheet of paraffined paper.

Removal of Specimens from Molds.

7. At the end of 48 hours the test specimens shall be removed from the molds and buried in damp sand except in case the molds shown in Fig. 3 are used, when they may be buried in damp sand without removal of the mold, thus permitting shipping in the molds

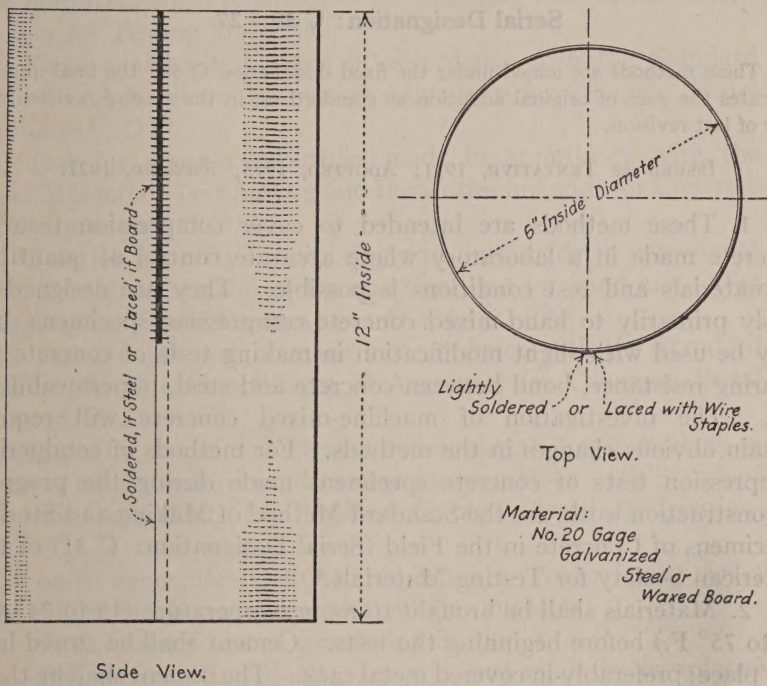
Soldered, if Steel or Laced, if Board
12" Inside
Side View.
6" Inside Diameter
Lightly Soldered or Laced with Wire Staples.
Top View.
Material:
No. 20 Gage Galvanized Steel or Waxed Board.

FIG. 3.

Storage of Specimens.

8. (*a*) The test specimens shall remain buried in damp sand until 10 days prior to the date of test. They shall then be well packed in damp sand or wet shavings and shipped to the testing laboratory, where they shall be stored either in a moist room or in damp sand until the date of test.

(*b*) Should a 7-day test be required, the test specimens shall remain at the work as long as possible to harden and then shall be shipped so as to arrive at the laboratory in time for test on the required date.

(*c*) Test specimens shall be protected from drying after removal from damp storage and before testing.

STANDARD METHODS
OF
MAKING COMPRESSION TESTS OF CONCRETE[1]

Serial Designation: C 39 – 27

These methods are issued under the fixed designation C 39; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED, 1925; REVISED, 1927.

Scope.

1. These methods are intended to cover compression tests of concrete made in a laboratory where accurate control of quantities of materials and test conditions is possible. They are designed to apply primarily to hand-mixed concrete compression specimens, but may be used with slight modification in making tests of concrete for wearing resistance, bond between concrete and steel, impermeability, etc. The investigation of machine-mixed concrete will require certain obvious changes in the methods. For methods of conducting compression tests of concrete specimens made during the progress of construction work, see the Standard Method of Making and Storing Specimens of Concrete in the Field (Serial Designation: C 31) of the American Society for Testing Materials.[2]

Preparation of Materials.

2. Materials shall be brought to room temperature (18 to 24° C., 65 to 75° F.) before beginning the tests. Cement shall be stored in a dry place; preferably in covered metal cans. The cement shall be thoroughly mixed in advance, in order that the sample may be uniform throughout the tests. It shall be sieved through a No. 16 (1190-micron) sieve and all lumps rejected. Aggregates shall be in a room-dry condition when used in concrete tests. In general, aggregates should be separated on the No. 4 (4760-micron), $\frac{3}{8}$-in. and $1\frac{1}{2}$ in. sieves and recombined to the average original sieve analysis for each batch. Fine aggregate should be separated into different sizes also, in cases where unusual gradings are being studied.

Sampling for Preliminary Tests.

3. Representative samples of all concrete materials shall be secured for preliminary tests prior to the proportioning and mixing of the concrete. Cement test samples may be made up of a small

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C–9 on Concrete and Concrete Aggregates.

[2] See p. 108.

quantity from each sack used in the concrete tests. Test samples of aggregates may be taken from larger lots by quartering.

Cement Tests.

4. Cement shall be tested in accordance with the methods described in the Standard Specifications and Tests for Portland Cement (Serial Designation: C 9) of the American Society for Testing Materials.[1]

Fine Aggregate Tests.

5. Fine aggregates (passing through a No. 4 (4760-micron) sieve) shall be subjected, when required, to the following tests of the American Society for Testing Materials:

(*a*) Sieve analysis test made in accordance with the Standard Method of Test for Sieve Analysis of Aggregates for Concrete (Serial Designation: C 41).[2]

(*b*) Test for organic impurities made in accordance with the Standard Method of Test for Organic Impurities in Sands for Concrete (Serial Designation: C 40).[3]

(*c*) Test for quantity of silt, clay and dust made in accordance with the Tentative Method of Decantation Test for Sand and Other Fine Aggregates (Serial Designation: D 136 – 22 T).[4]

(*d*) Test for unit weight made in accordance with the Standard Method of Test for Unit Weight of Aggregate for Concrete (Serial Designation: C 29).[5]

(*e*) Strength test of 1:3 mortar by weight at 7 and 28 days in comparison with standard sand mortar in accordance with Standard Specifications and Tests for Portland Cement (Serial Designation: C 9).[1]

Coarse Aggregate Tests.

6. Coarse aggregates (retained on a No. 4 (4760-micron) sieve) shall be subjected when required to the following tests:

(*a*) Sieve analysis test as specified in Section 5 (*a*);

(*b*) Test for quantity of silt, clay and dust made in accordance with Standard Method of Test for Quantity of Clay and Silt in Gravel for Highway Construction (Serial Designation: D 72) of the American Society for Testing Materials.[6]

(*c*) Test for unit weight as specified in Section 5 (*d*).

Mixed Aggregate Tests.

7. The unit weight of mixed fine and coarse aggregates as used in concrete tests shall be determined in accordance with the method specified in Section 5 (*d*).

Proportioning.

8. The quantities of each size of aggregate to be used in each batch shall be determined on the basis of the sieve analysis and the unit weight of the mixed aggregate. The exact quantities of cement

[1] See p. 23. [2] See p. 124. [3] See p. 123.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part I, p. 802 (1922); also 1927 Book of A.S.T.M. Tentative Standards, p. 501.

[5] See p. 120. [6] See p. 472.

and of each size of aggregate for each batch shall be determined by weight. The quantity of water for each batch shall be accurately measured. The quantities of materials may be expressed as (*a*) 1 volume of cement to .... volumes of total aggregate mixed as used, or (*b*) 1 volume of cement, ... volumes of fine aggregate, and .... volumes of coarse aggregate, or (*c*) 1 part by weight of cement .... parts by weight of fine aggregate, and .... parts by weight of coarse aggregate.

NOTE.—It is impracticable to give a general method for proportioning concrete for experimental purposes; the details will necessarily vary widely with the purpose for which the tests are made. The following procedure is suggested for specific cases:

(*a*) Vary the cement content by 10-per-cent intervals above and below assumed quantity.

(*b*) Vary the proportions of fine to coarse aggregate, measured separately, at intervals of 10 per cent.

(*c*) Vary the quantity of mixing water by intervals of 10 per cent.

Size and Shape of Specimens.

9. Compression tests of concrete shall be made on cylinders of diameter equal to one-half the length. The standard shall be 6 by 12-in. cylinders where the coarse aggregate does not exceed 2-in. in size; for aggregates larger than 2 in., 8 by 16-in. cylinders shall be used; 2 by 4-in. cylinders may be used for mixtures without coarse aggregate.

Mixing Concrete.

10. (*a*) Concrete shall be mixed by hand in batches of such size as to leave a small quantity of concrete after molding a single test specimen. The batch shall preferably be mixed in a shallow metal pan with a 10-in. bricklayer's trowel which has been blunted by cutting off about $2\frac{1}{2}$ in. of the point, as follows:

(*b*) The cement and fine aggregate shall be mixed dry until the mixture is homogeneous in color;

(*c*) The coarse aggregate shall be added and mixed dry;

(*d*) Sufficient water shall be added to produce concrete of the required workability;

NOTE.—Concrete tests should be made on plastic mixtures. It is of the utmost importance that a uniform degree of workability be secured in tests involving comparisons of different materials and methods.

(*e*) The mass shall be mixed thoroughly until the resulting concrete is homogeneous in appearance.

Workability.

11. The workability or plasticity of each batch of concrete shall be measured immediately after mixing by one of the following methods:

(*a*) Slump test made in accordance with the Tentative Method of Test for Consistency of Portland-Cement Concrete (Serial Designation: D 138 – 26 T) of the American Society for Testing Materials.[1]

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I, p. 874 (1926); also 1927 Book of A.S.T.M. Tentative Standards, p. 503.

(*b*) Flow test made by placing a metal mold in the shape of a frustum of a cone $6\frac{3}{4}$ in. in top diameter, 10 in. in bottom diameter, 5 in. in depth, on the table of the flow apparatus.[1] The fresh concrete shall be placed in the mold in two layers. Each layer shall be puddled as described in Section 13. Immediately after molding, the surplus concrete shall be struck off with a trowel and the mold shall be removed by a steady upward pull; the table raised $\frac{1}{2}$ in. and dropped 15 times in about 10 seconds by means of a suitable cam and crank. The spread of the fresh concrete due to this treatment expressed as a percentage of the original bottom diameter of the cone is the "flow."

Molds.

12. The molds shall preferably be of metal. Each mold shall be provided with a machined metal base plate, and shall be oiled with a heavy mineral oil before using. Particular care shall be taken to obtain tight molds so that the mixing water will not escape during molding.

NOTE.—The best type of mold consists of lengths of cold-drawn steel tubing, split along one element and closed by means of a circumferential band and bolt. Satisfactory molds can be made from lengths of steel water-pipe machined on the inside, from rolled metal plates, from galvanized steel, machined iron or steel castings. Paraffined cardboard molds will give good results under expert supervision.

Molding Test Specimens.

13. Concrete test specimens shall be molded by placing the fresh concrete in the mold in layers 3 to 4 in. in thickness. Each layer shall be puddled with 25 strokes with a $\frac{5}{8}$-in. rod 24 in. in length, bullet-pointed at the lower end. After the top layer has been puddled, the surplus concrete shall be struck off with a trowel, and the mold covered with a piece of plate glass at least $\frac{1}{4}$ in. in thickness or a machined metal plate which will be used later in capping the test specimen.

Capping Specimens.

14. Two to four hours after molding, the test specimens shall be capped with a thin layer of stiff neat-cement paste in order that the cylinder may present a smooth end for testing. The cap shall be formed by means of a piece of plate glass $\frac{1}{4}$ in. in thickness or a machined metal plate $\frac{1}{2}$ in. in thickness and of a diameter 2 or 3 in. larger than that of the mold. The plate shall be worked on the fresh cement paste until it rests on top of the mold. The cement for capping should be mixed to a stiff paste 2 to 4 hours before it is to be used in order to avoid the tendency of the cap to shrink. Adhesion of the concrete to the top and bottom plates may be avoided by oiling the plates or by inserting a sheet of paraffined paper.

Curing Test Specimens.

15. Concrete test specimens shall be removed from the molds 20 to 48 hours after molding, marked, weighed and stored in damp

[1] For a description and illustration of one design for a flow-table, see *Proceedings*, Am. Soc. Testing Mats., Vol. XX, Part II, p. 242 (1920), and "Concrete," June, 1920, p. 274.

sand, under damp cloths or in a moist chamber until the date of test. The temperature of the curing room should not fall outside the range of 18 to 24° C. (60 to 75° F.).

Age at Test. 16. Tests shall be made at the age of 7 and 28 days; ages of 3 months and 1 year are recommended, if longer-time tests are required.

Sequence of Tests. 17. Three to five test specimens should be made on different days in investigations in which accurate comparisons are desired.

Preparation of Tests. 18. Compression tests shall be made immediately upon removal of the concrete test specimens from the curing room; that is, the test specimens shall be loaded in a damp condition. The length and average diameter of the test specimen shall be measured in inches and hundredths; two diameters shall be measured at right angles near the mid-length. The test specimen shall be weighed immediately before testing.

Method of Testing. 19. In general, only the ultimate compressive strength of the cylinders need be observed. The metal bearing plates of the testing machine shall be placed in contact with the ends of the test specimen; cushioning materials shall not be used. An adjustable bearing block shall be used to transmit the load to the test specimen. The bearing block shall be used on *top* of the specimen in vertical testing machines. The diameter of the bearing block shall be the same or slightly larger than that of the test specimen. The upper or lower section of the bearing block shall be kept in motion as the head of the testing machine is brought to a bearing on the test specimen.

Application of Load. 20. The load shall be applied uniformly and without shock. The moving head of the testing machine should travel at the rate of about 0.05 in. per minute when the machine is running idle.

Record of Tests. 21. The total load indicated by the testing machine at failure of the test specimen shall be recorded and the unit compressive strength calculated in pounds per square inch, the area computed from the average diameter of the cylinder being used. The type of failure and appearance of the concrete shall be noted.

Weight of Concrete. 22. The weight of the concrete in pounds per cubic foot shall be determined from the weight of the specimens and their dimensions.

Density and Yield. 23. Density and yield of concrete when required shall be calculated from the unit volumes of the constituent materials and the volume of the fresh or set concrete as desired. Density is here understood to be the ratio of solids in the concrete to the total volume of the mass. Yield is the volume of concrete resulting from the volume of aggregate mixed as used.

24. The report shall include the following: Report.

(*a*) The kind and origin of concrete materials;

(*b*) Complete data on all tests of cement and aggregates;

(*c*) A description of methods of making and testing the concrete, where methods deviate from the proposed standards;

(*d*) The quantities of cement, aggregates and water in each batch;

(*e*) The method of measuring workability or plasticity with "slump" or "flow" of concrete;

(*f*) The quantity of water expressed as a ratio to volume of cement;

(*g*) The age at test;

(*h*) The size of test specimens;

(*i*) The date of molding and testing each cylinder;

(*j*) The compressive strength in pounds per square inch of each test specimen and the average of tests in a set;

(*k*) A description of failure and appearance of concrete on each test specimen;

(*l*) The unit weight, density and yield of the concrete.

STANDARD METHODS
OF
SECURING SPECIMENS OF HARDENED CONCRETE FROM THE STRUCTURE[1]

Serial Designation: C 42 – 27

These methods are issued under the fixed designation C 42; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1927.

Scope.

1. These methods and precautions are intended to apply to securing test specimens from hardened concrete in the structure.

Precautions.

2. A specimen from hardened concrete to be tested for strength shall not be taken until the concrete has become hard enough so that the cutting of the specimen will not disturb the bond between the mortar and the coarse aggregate of the specimen.

Apparatus.

3. The specimen from the structure can best be secured by use of a core drill. For specimens taken perpendicular to a horizontal surface, a drill using chilled shot may be used; but when taken perpendicular to a vertical surface, a diamond drill should be used.

Size and Shape of Specimens.

4. (*a*) The core specimen taken shall be as nearly as possible a cylinder whose length is twice the diameter.

(*b*) In securing a specimen perpendicular to a horizontal surface care shall be taken to secure, if possible, a specimen whose beds shall be parallel to the horizontal bed of the concrete as originally placed.

(*c*) In securing a specimen perpendicular to a vertical surface, or to a surface with a batter, care shall be taken as to the place of securing the specimen. The lower portion of any one unit of depositing in mass concrete is more dense than is the upper portion of the unit, therefore a specimen shall be taken from near the middle of such unit of deposit.

Procedure.

5. A specimen to be taken from hardened concrete which has been removed from a structure shall be cut out by a drill or shall be cut into a symmetrical test-piece by tooling or by sawing. The method of sawing can seldom be used to advantage in the field. In selecting

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

this test specimen care shall be used to see that the concrete selected has not been injured or shattered by the method of its removal from the structure.

Ends of Test Specimens.

6. The specimen secured shall have ends, or beds, as nearly as possible perpendicular to its axis, and, if the specimen has ends with uneven surfaces, these ends shall be made to parallel plane surfaces by capping with a thin layer of mortar richer than the mortar of the specimen or with a mixture of cement and calcined gypsum (plaster of Paris).

Strength Correction Factor.

7. In case the dimensions of a specimen are necessarily appreciably different from the recommended shape which requires a height twice the diameter, allowance for the ratio of height to diameter must be made in determining the strength. When the height is more than twice the diameter, that is, when the ratio is greater than two, no correction need be made. For other ratios corrections shall be made as indicated in the following table. Values not given in the table shall be determined by interpolation:

Ratio of Height of Cylinder to Diameter, $\left(\frac{h}{d}\right)$	
1.75	multiply crushing strength by 0.98
1.50	" " " " 0.95
1.25	" " " " 0.94
1.10	" " " " 0.90
1.00	" " " " 0.85
0.75	" " " " 0.70
0.50	" " " " 0.50

Test Precautions.

8. The direction of the application of the load in the compression test shall be stated with reference to the direction of the compaction of the mass of concrete in the structure, and where it is practicable the compression load shall be applied in the same direction. In order that the tests be made under uniform conditions as to moisture content, the test specimens shall be completely submerged in water for 48 hours and the compression test shall be made immediately thereafter.

STANDARD METHOD OF TEST
FOR
UNIT WEIGHT OF AGGREGATE FOR CONCRETE[1]

Serial Designation: C 29 – 27

This method is issued under the fixed designation C 29; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1920; ADOPTED, 1921; REVISED, 1927.

Scope.

1. This method is intended to cover the determination of unit weight of fine, coarse, or mixed aggregate for concrete.

Apparatus.

2. (*a*) The apparatus required consists of a cylindrical metal measure, a tamping rod, and a scale or balance, sensitive to 0.5 per cent of the weight of the sample to be weighed.

(*b*) *Measures.*—The measure shall be of metal, preferably machined to accurate dimensions on the inside, cylindrical in form, water-tight, and of sufficient rigidity to retain its form under rough usage, with top and bottom true and even, and preferably provided with handles.

The measure shall be of $\frac{1}{10}$, $\frac{1}{2}$ or 1-cu.-ft. capacity, depending on the maximum diameter of the coarsest particles in the aggregate, and shall be of the following dimensions:

Capacity, cu. ft.	Inside Diameter, in.	Inside Height, in.	Minimum Thickness of Metal, U. S. Gage	Diameter of Largest Particles of Aggregate, in.
$\frac{1}{10}$	6.00	6.10	No. 11	Under $\frac{1}{2}$
$\frac{1}{2}$	10.00	11.00	No. 8	Under $1\frac{1}{2}$
1	14.00	11.23	No. 5	Over $1\frac{1}{2}$

(*c*) *Tamping Rod.*—The tamping rod shall be a straight metal rod $\frac{5}{8}$ in. in diameter and 24 in. in length, with one end tapered for a distance of 1 in. to a blunt bullet-shaped point.

Calibrating the Measure.

3. The measure shall be calibrated by accurately determining the weight of water at 16.7° C. (62° F.) required to fill it. The factor for

[1] Under the standardization procedure of the Society, this method is under jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

any unit shall be obtained by dividing the unit weight of water at 16.7° C. (62° F.) (62.355 lb. per cu. ft.) by the weight of water at 16.7° C. (62° F.) required to fill the measure.

4. The sample of aggregate shall be room dry and thoroughly mixed. **Condition of Sample.**

5. (*a*) The measure shall be filled one-third full and the top levelled off with the fingers. The mass shall be puddled with the tamping rod with 25 strokes, evenly distributed over the surface. The measure shall be filled two-thirds full and again puddled with 25 strokes as before. The measure shall then be filled to overflowing, puddled 25 times, and the surplus aggregate struck off, using the tamping rod as a straight edge. **Method.**

In puddling the first layer the rod should not be permitted to forcibly strike the bottom of the measure. In puddling the second and final layers, only enough force to cause the tamping rod to penetrate the last layer of aggregate placed in the measure should be used.

(*b*) The net weight of the aggregate in the measure shall be determined. The unit weight of the aggregate shall then be obtained by multiplying the net weight of the aggregate by the factor found as described in Section 3.

6. Results with the same sample should check within 1 per cent. **Accuracy.**

STANDARD METHOD OF TEST

FOR

DETERMINATION OF VOIDS IN FINE AGGREGATE FOR CONCRETE[1]

Serial Designation: C 30 – 22

This method is issued under the fixed designation C 30; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1920; ADOPTED, 1922.

This method was approved May 29, 1923, as "Tentative American Standard" by the American Engineering Standards Committee.

Formula for Voids.

1. The voids in fine aggregate for cement concrete shall be determined by the formula:

$$\text{Percentage of Voids} = \frac{(\text{sp. gr.} \times 62.355) - \text{Wt.}}{\text{sp. gr.} \times 62.355} \times 100$$

Symbol "sp. gr."

2. The symbol "sp. gr." represents the Apparent Specific Gravity of the fine aggregate as determined by the Standard Method of Test for Apparent Specific Gravity of Sand, Stone and Slag Screenings, and Other Fine Non-Bituminous Highway Materials (Serial Designation: D 55) of the American Society for Testing Materials.[2]

Weight of Water.

3. The quantity 62.355 is the weight in pounds of one cubic foot of water at the standard temperature of 16.7° C. (62° F.).

Symbol "Wt."

4. The symbol "Wt." represents the weight in pounds per cubic foot of the fine aggregate as determined by the Standard Method of Test for Unit Weight of Aggregate for Concrete (Serial Designation: C 29) of the American Society for Testing Materials.[3]

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] See p. 468.] [3] See p. 120.

STANDARD METHOD OF TEST
FOR
ORGANIC IMPURITIES IN SANDS FOR CONCRETE[1]

Serial Designation: C 40 – 27

This method is issued under the fixed designation C 40; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED, 1922; REVISED, 1927.

Scope.

1. This method of test is an approximate method of determining the presence of injurious organic compounds in natural sands which are to be used in cement mortar or concrete. The principal value of the test is to furnish a warning that further tests of the sands are necessary before they are approved for use.

Sample.

2. A representative test sample of sand weighing about one pound shall be obtained by quartering or by the use of a sampler.

Method.

3. (*a*) A 12-oz. graduated clear glass bottle shall be filled to the $4\frac{1}{2}$-oz. mark with the sand to be tested.

(*b*) A 3-per-cent solution of sodium hydroxide in water shall be added until the volume of the sand and liquid indicated after shaking is 7 liquid ounces.

(*c*) The bottle shall be stoppered, shaken vigorously and then allowed to stand for 24 hours.

Standard Color Solution.

4. A standard color solution shall be prepared by adding 2.5 cc. of a 2-per-cent solution of tannic acid in 10-per-cent alcohol to 97.5 cc. of a 3-per-cent sodium hydroxide solution. This shall be placed in a 12-oz. bottle, stoppered, shaken vigorously, and allowed to stand for 24 hours.

Determination of Color Value.

5. (*a*) After standing 24 hours, the color of the clear liquid above the sand shall be compared with the color of the standard color solution prepared at the same time and in accordance with the method described in Section 4, or with a glass of color similar to the color of the standard solution.

(*b*) Solutions darker in color than the standard color have a "color value" higher than 250 parts per million in terms of tannic acid.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

STANDARD METHOD OF TEST
FOR
SIEVE ANALYSIS OF AGGREGATES FOR CONCRETE[1]

Serial Designation: C 41 – 24

This method is issued under the fixed designation C 41; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED, 1922; REVISED, 1924.

Sampling. 1. A representative test sample of the aggregate shall be selected by quartering or by use of a sampler, which after drying will give not less than the following:

(*a*) Fine aggregate, 500 g.

(*b*) Coarse aggregate or a mixture of fine and coarse aggregates, weight in grams, 3000 times the size of the largest sieve required, measured in inches.

TABLE I.—REQUIREMENTS FOR SIEVE OPENINGS AND WIRE DIAMETERS WITH PERMISSIBLE VARIATIONS.[2]

Sieve Number or Size in inches	Sieve Opening		Wire Diameter		Tolerance, per cent			
	mm.	in.	mm.	in.	Average Opening	Wire Diameter		Maximum Opening
						Under	Over	
No. 100 (149-micron)	0.149	0.0059	0.102	0.0040	±6	15	35	40
No. 50 (297-micron)	0.297	0.0117	0.188	0.0074	±6	15	35	40
No. 30 (590-micron)	0.59	0.0232	0.33	0.0130	±5	15	30	25
No. 16 (1190-micron)	1.19	0.0469	0.54	0.0213	±3	15	30	10
No. 8 (2380-micron)	2.38	0.0937	0.84	0.0331	±3	15	30	10
No. 4 (4760-micron)	4.76	0.187	1.27	0.050	±3	15	30	10
⅜-in	9.5	0.375	2.33	0.092	±3	10	10	10
¾-in	19.0	0.75	3.42	0.135	±3	10	10	10
1-in	25.4	1.00	4.12	0.162	±3	10	10	10
1½-in	38.0	1.50	4.50	0.177	±3	10	10	10
2-in	50.8	2.00	4.88	0.192	±3	10	10	10
3-in	76.0	3.00	6.3	0.25	±3	10	10	10

Treatment of Sample. 2. The sample shall be dried to constant weight at a temperature not exceeding 110° C. (230° F.).

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] The dimensions and tolerances for these sieves conform to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

3. (*a*) The sieves shall be of square-mesh wire-cloth and shall be mounted on substantial frames constructed in a manner that will prevent loss of material during sieving. **Sieves.**

(*b*) The size of wire and sieve openings shall conform to the requirements given in Table I.

4. (*a*) The sample shall be separated into a series of sizes by means of the sieves specified in Section 3. Sieving shall be continued until not more than 1 per cent by weight of the residue passes any sieve during 1 minute. **Procedure.**

(*b*) Each size shall be weighed on a balance or scale which is sensitive to 0.001 of the weight of the test sample.

(*c*) The percentage by weight of the total sample which is finer than each of the sieves shall be computed.

5. (*a*) The percentages in sieve analysis shall be reported to the nearest whole number. **Report.**

(*b*) If more than 15 per cent of a fine aggregate is coarser than the No. 4 (4760-micron) sieve, or more than 15 per cent of a coarse aggregate is finer than the No. 4 (4760-micron) sieve, the sieve analysis of the portions finer and coarser than this sieve shall be reported separately.

STANDARD SPECIFICATIONS
FOR
PAVING BRICK[1]

Serial Designation: C 7–15

These specifications are issued under the fixed designation C 7; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1915.

The quality and acceptability of paving brick, in the absence of other special tests mutually agreed upon in advance by the seller on the one side and the buyer on the other side shall be determined by the following procedure:

I. *The Rattler Test*, for the purpose of determining whether the material as a whole possesses to a sufficient degree strength, toughness and hardness.

II. *Visual Inspection*, for the purpose of determining whether the physical properties of the material as to dimensions, accuracy and uniformity of shape and color, are in general satisfactory, and for the purpose of culling out from the shipment individually imperfect or unsatisfactory brick.

The acceptance of paving brick as satisfactorily meeting one of these tests shall not be construed as in any way waiving the other.

I. THE RATTLER TEST.

THE SELECTION OF SAMPLES FOR TEST.

1. *Place of Sampling.*—In general, where a shipment of bricks involving a quantity of less than 100,000 is under consideration, the sampling may be done either at the brick factory prior to shipment, or on cars at their destination or on the street, when delivered ready for use. When the quantity under consideration exceeds 100,000 the sampling shall be done at the factory prior to shipment. Bricks accepted as the result of test prior to shipment, shall not be liable to subsequent rejection as a whole, but are subject to such culling as is provided for under Part II, Visual Inspection.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-3 on Brick.

2. *Method of Selecting Samples.*—In general, the buyer shall select his own samples from the material which the seller proposes to furnish. The seller shall have the right to be present during the selection of a sample. The sampler shall endeavor, to the best of his judgment, to select brick representing the average of the lot. No samples shall include bricks which would be rejected by visual inspection as provided in Part II, except that where controversy arises, whole tests may be selected to determine the admissibility of certain types or portions of the lot having a characteristic appearance in common. In cases where prolonged controversy occurs between buyer and seller and samples selected by each party fail to show reasonable concurrence, then both parties shall unite in the selection of a disinterested person to select the samples, and both parties shall be bound by the results of samples thus selected.

3. *Number of Samples per Lot.*—In general, one sample of ten bricks shall be tested for every 10,000 bricks contained in the lot under consideration; but where the total quantity exceeds 100,000, the number of samples tested may be fewer than one per 10,000, provided that they shall be distributed as uniformly as practicable over the entire lot.

4. *Shipment of Samples.*—Samples which must be transported long distances by freight or express shall be carefully put up in packages holding not more than twelve bricks each. When more than six bricks are shipped in one package, it shall be so arranged as to carry two parallel rows of bricks side by side, and these rows shall be separated by a partition. In event of some of the bricks being cracked or broken in transit, the sample shall be disqualified if there are not remaining ten sound undamaged bricks.

5. *Storage and Care of Samples.*—Samples shall be carefully handled to avoid breakage or injury. They shall be kept in the dry so far as practicable. If wet when received, or known to have been immersed or subjected to recent prolonged wetting, they shall be dried for at least six hours in a temperature of 100° F. before testing.

THE CONSTRUCTION OF THE RATTLER.

6. *General Design.*—The machine shall be of good mechanical construction, self-contained, and shall conform to the following details of material and dimensions, and shall consist of barrel, frame, and driving mechanism as herein described.

Accompanying these specifications is a complete drawing of a rattler which will meet the requirements, and to which reference should be made (Plate I).

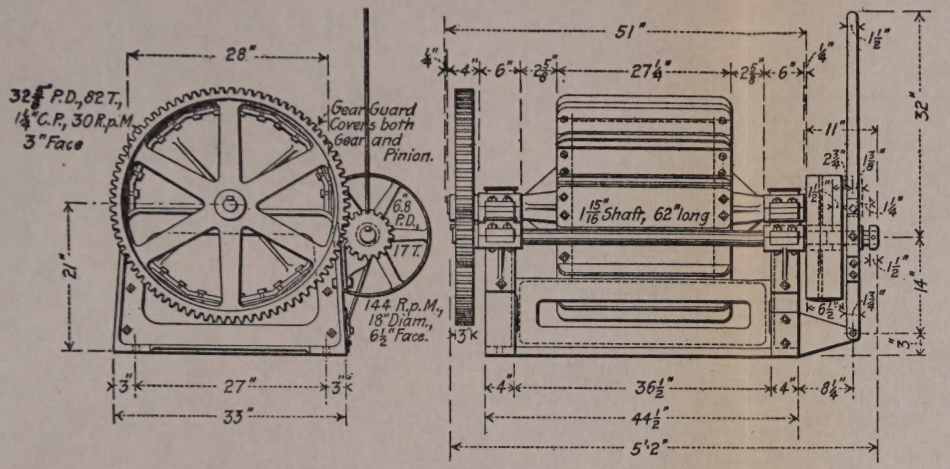
7. *The Barrel.*—The barrel of the machine shall be made up of the heads, headliners, staves and stave-liners.

The heads may be cast in one piece with the trunnions, which shall be $2\frac{1}{2}$ in. in diameter, and shall have a bearing 6 in. in length, or they may be cast with heavy hubs, which shall be bored out for $2\frac{7}{16}$-in. shafts, and shall be keyseated for two keys, each $\frac{1}{2}$ by $\frac{3}{8}$ in. and spaced 90 deg. apart. The shaft shall be a snug fit and when keyed shall be entirely free from lost motion. The distance from the end of the shaft or trunnion to the inside face of the head shall be $15\frac{3}{8}$ in. in the head for the driving end of the rattler, and $11\frac{3}{8}$ in. for the other head, and the distance from the face of the hubs to the inside face of the heads shall be $5\frac{1}{8}$ in.

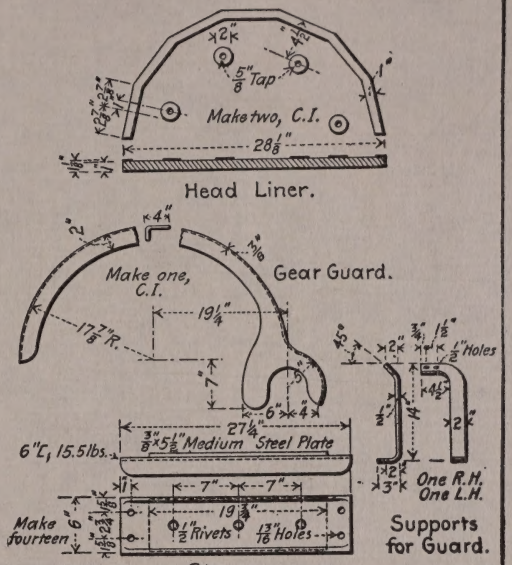
The heads shall be not less than $\frac{3}{4}$ in. thick, nor more than $\frac{7}{8}$ in. thick. In outline, each head shall be a regular 14-sided polygon inscribed in a circle $28\frac{3}{8}$ in. in diameter. Each head shall be provided with flanges not less than $\frac{3}{4}$ in. thick and extending outward $2\frac{1}{2}$ in. from the inside face of the head to afford a means of fastening the staves. The surface of the flanges of the head shall be smooth and give a true and uniform bearing for the staves. To secure the desired true and uniform bearing the surfaces of the flanges of the head shall be either ground or machined. The flanges shall be slotted on the outer edge, so as to provide for two $\frac{3}{4}$-in. bolts at each end of each stave, said slots to be $\frac{13}{16}$ in. wide and $2\frac{3}{4}$ in., center to center. Each slot shall be provided with a recess for the bolt head, which shall act to prevent the turning of the same. Between each two slots there shall be a brace $\frac{3}{8}$ in. thick, extending down the outward side of the head not less than 2 in.

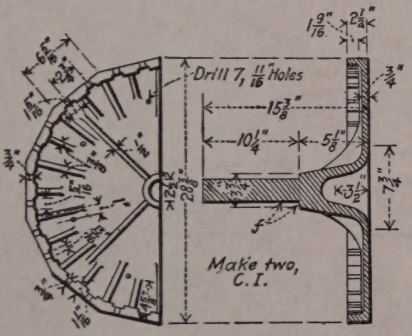
There shall be for each head a cast-iron headliner 1 in. in thickness and conforming to the outline of the head, but inscribed in a circle $28\frac{1}{8}$ in. in diameter. This headliner shall be fastened to the head by seven $\frac{5}{8}$ in. cap-screws, through the head from the outside. Whenever these headliners become worn down $\frac{1}{2}$ in. below their initial surface level at any point of their surface, they shall be replaced with new ones. The metal of these headliners shall be hard machinery iron and should contain not less than one per cent of combined carbon.

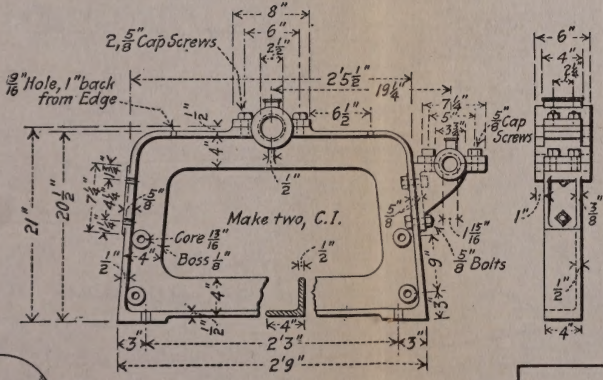
The staves shall be made of 6-in. medium-steel structural

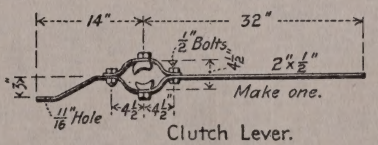


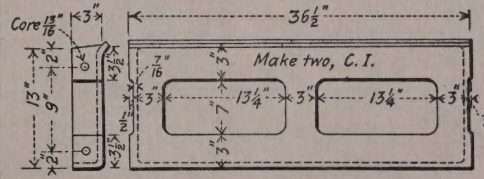
End Elevation. Side Elevation.

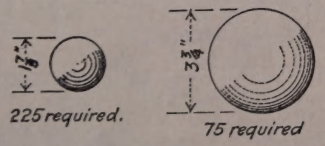










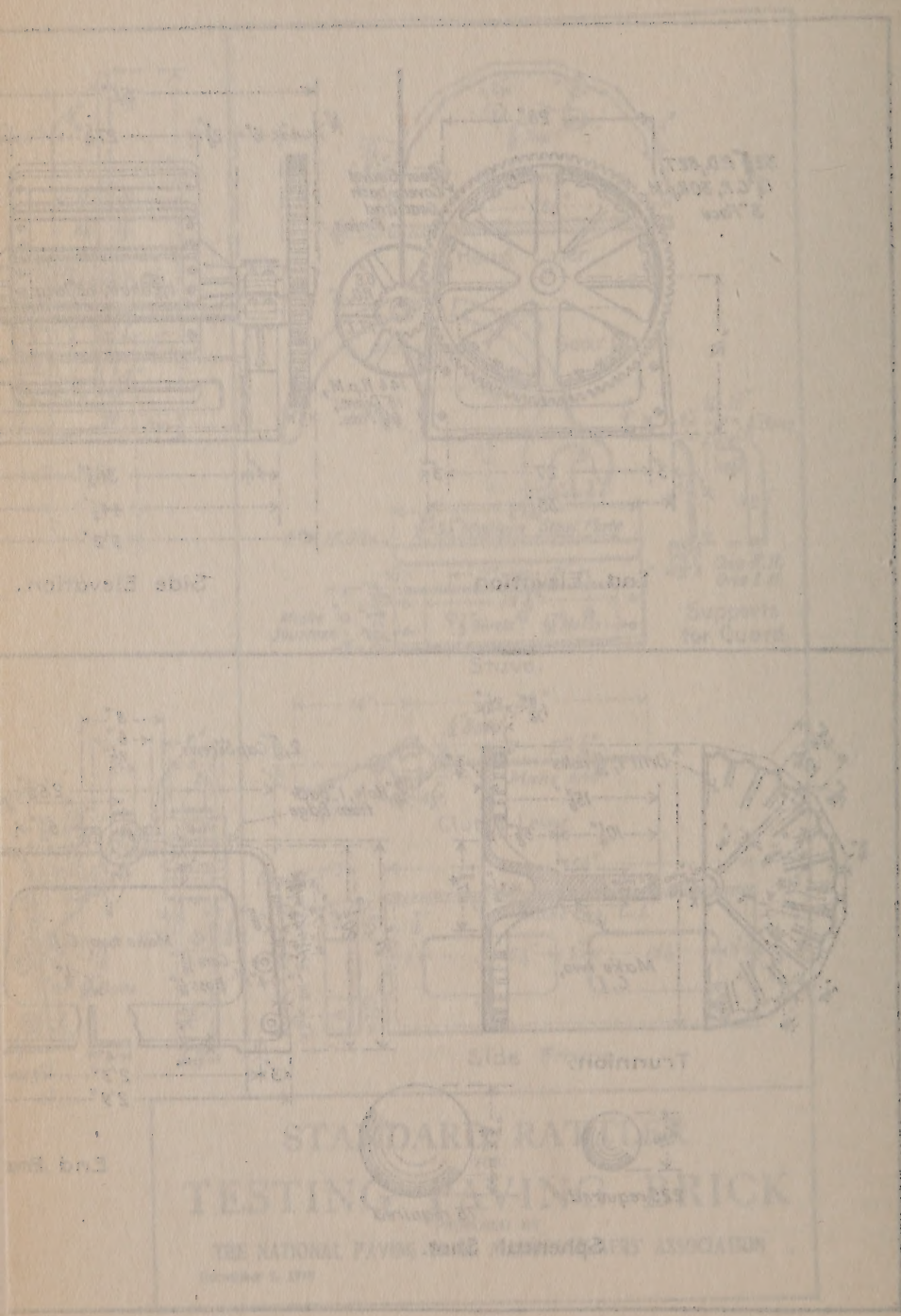


STANDARD RATTLER FOR TESTING PAVING BRICK

PROPOSED BY

THE NATIONAL PAVING BRICK MANUFACTURERS' ASSOCIATION

December 1, 1910



channels, $27\frac{1}{4}$ in. long and weighing 15.5 lb. per lineal foot. The staves shall have two holes $\frac{13}{16}$ in. in diameter, drilled in each end, the center line of the holes being 1 in. from the end and $1\frac{3}{8}$ in. either way from the longitudinal center line. The spaces between the staves shall be as uniform as practicable, but shall not exceed $\frac{5}{16}$ in.

The interior or flat side of each stave shall be protected by a liner $\frac{3}{8}$ in. thick by $5\frac{1}{2}$ in. wide by $19\frac{3}{4}$ in. long. The liner shall consist of medium-steel plate, and shall be riveted to the channel by three $\frac{1}{2}$-in. rivets, one of which shall be on the center line both ways and the other two on the longitudinal center line and spaced 7 in. from the center each way. The rivet holes shall be countersunk on the face of the liner and the rivets shall be driven hot and chipped off flush with the surface of the liners. These liners shall be inspected from time to time, and if found loose shall be at once re-riveted.

Any test at the expiration of which a stave-liner is found detached from the stave or seriously out of position shall be rejected. When a new rattler, in which a complete set of new staves is furnished, is first put into operation, it shall be charged with 400 lb. of shot of the same sizes, and in the same proportions as provided in Section 9, and shall then be run for 18,000 revolutions at the usual prescribed rate of speed. The shot shall then be removed and a standard shot charge inserted, after which the rattler may be charged with brick for a test.

No stave shall be used for more than 70 consecutive tests without renewing its lining. Two of the 14 staves shall be removed and relined at a time in such a way that of each pair, one falls upon one side of the barrel and the other upon the opposite side, and also so that the staves changed shall be consecutive but not contiguous; for example, 1 and 8, 3 and 10, 5 and 12, 7 and 14, 2 and 9, 4 and 11, 6 and 13, etc., to the end that the interior of the barrel at all times shall present the same relative condition of repair. The changes in the staves should be made at the time when the shot charges are being corrected, and the record must show the number of charges run since the last pair of new lined staves was placed in position.

The staves when bolted to the heads shall form a barrel 20 in. long, inside measurement, between head liners. The liners of the staves shall be so placed as to drop between the head-liners. The staves shall be bolted tightly to the heads by four $\frac{3}{4}$-in. bolts, and each bolt shall be provided with a lock nut,

and shall be inspected at not less frequent intervals than every fifth test and all nuts kept tight. A record shall be made after each inspection showing in what condition the bolts were found.

8. *The Frame and Driving Mechanism.*—The barrel shall be mounted on a cast-iron frame of sufficient strength and rigidity to support it without undue vibration. It shall rest on a rigid foundation with or without the interposition of wooden plates, and shall be fastened thereto by bolts at not less than four points.

It shall be driven by gearing whose ratio of driver to driven is not less than one to four. The counter shaft upon which the driving pinion is mounted shall not be less than $1\frac{15}{16}$ in. in diameter, with bearing not less than 6 in. in length. If a belt drive is used the pulley shall not be less than 18 in. in diameter and $6\frac{1}{2}$ in. in face. A belt at least 6 in. in width properly adjusted, to avoid unnecessary slipping, should be used.

9. *The Abrasive Charge.*—The abrasive charge shall consist of cast-iron spheres of two sizes. When new, the larger spheres shall be 3.75 in. in diameter and shall weigh approximately 7.5 lb. (3.40 kg.) each. Ten spheres of this size shall be used.

These shall be weighed separately after each ten tests, and if the weight of any large sphere falls to 7 lb. (3.175 kg.) it shall be discarded and a new one substituted; provided, however, that all of the large spheres shall not be discarded and substituted by new ones at any single time, and that so far as possible the large spheres shall compose a graduated series in various stages of wear.

When new, the smaller spheres shall be 1.875 in. in diameter and shall weigh approximately 0.95 lb. (0.43 kg.) each. In general, the number of small spheres in a charge shall not fall below 245 nor exceed 260. The collective weight of the large and small spheres shall be as nearly 300 lb. as possible. No small sphere shall be retained in use after it has been worn down so that it will pass a circular hole 1.75 in. in diameter, drilled in an iron plate $\frac{1}{4}$ in. in thickness, or weigh less than 0.75 lb. (0.34 kg.). Further, the small spheres shall be tested, by passing them over the above plate or by weighing, after every ten tests, and any which pass through or fall below the specified weight, shall be replaced by new spheres; provided, further, that all of the small spheres shall not be rejected and

replaced by new ones at any one time, and that so far as possible the small spheres shall compose a graduated series in various stages of wear. At any time that any sphere is found to be broken or defective it shall at once be replaced.

The iron composing these spheres shall have a chemical composition within the following limits:

Combined carbon	not under	2.50	per cent	
Graphitic carbon	" over	0.25	"	
Silicon	" "	1.00	"	
Manganese	" "	0.50	"	
Phosphorus	" "	0.25	"	
Sulfur	" "	0.08	"	

For each new batch of spheres used, the chemical analysis shall be furnished by the maker or be obtained by the user, before introducing into the charge, and unless the analysis meets the above requirements, the batch of spheres shall be rejected.

THE OPERATION OF THE TEST.

10. *The Brick Charge.*—The number of bricks per test shall be ten for all bricks of so-called "block-size," whose dimensions fall between 8 and 9 in. in length, 3 and $3\frac{3}{4}$ in. in breadth, and $3\frac{3}{4}$ and $4\frac{1}{4}$ in. in thickness.[1] No brick should be selected as part of a regular test that would be rejected by any other requirements of the specifications under which the purchase is made.

11. *Speed and Duration of Revolution.*—The rattler shall be rotated at a uniform rate of not less than 29.5 nor more than 30.5 revolutions per minute, and 1800 revolutions shall constitute the test. A counting machine shall be attached to the rattler for counting the revolutions. A margin not to exceed ten revolutions will be allowed for stopping. Only one start and stop per test is generally acceptable. If, from accidental causes, the rattler is stopped and started more than once during a test, and the loss exceeds the maximum permissible under the specifications, the test shall be disqualified and another made.

12. *The Scales.*—The scales must have a capacity of not less than 300 lb., and must be sensitive to 0.5 oz., and must be tested by a standard test weight at intervals of not less than every ten tests.

[1] Where brick of larger or smaller sizes than the dimensions given above for blocks are to be tested, the same number of bricks per charge should be used, but allowance for the difference in size should be made in setting the limits for average and maximum rattler loss.

13. *The Results.*—The loss shall be calculated in percentage of the initial weight of the brick composing the charge. In weighing the rattled brick, any piece weighing less than 1 lb. shall be rejected.

14. *The Records.*—A complete and continuous record shall be kept of the operation of all rattlers working under these specifications. This record shall contain the following data concerning each test made:

1. The name of the person, firm or corporation furnishing each sample tested.
2. The name of the maker of the brick represented in each sample tested.
3. The name of the street, or contract, which the sample represented.
4. The brands or marks upon the bricks by which they were identified.
5. The number of bricks furnished.
6. The date on which they were received for test.
7. The date on which they were tested.
8. The drying treatment given before testing, if any.
9. The length, breadth and thickness of the bricks.
10. The collective weight of the ten large spherical shot used in making the test at the time of their last standardization.
11. The number and collective weight of the small spherical shot used in making the test, at the time of their last standardization.
12. The total weight of the shot charge, after its last standardization.
13. Certificate of the operator that he examined the condition of the machine as to staves, liners, and any other parts affecting the barrel, and found them right at the beginning of the test.
14. Certificate of the operator of the number of charges tested since the last standardization of shot charge and last renewals of stave liners.
15. The time of the beginning and ending of each test, and the number of revolutions made by the barrel during the test, as shown by the indicator.
16. Certificate of the operator as to number of stops and starts made in each test.
17. The initial collective weight of the ten bricks composing the charge and their collective weight after rattling.
18. The loss calculated in percentage of the initial weight; and the calculation itself.
19. The number of broken bricks and remarks upon the portions which were included in the final weighing.
20. General remarks upon the test and any irregularities occurring in its execution.
21. The date upon which the test was made.
22. The location of the rattler and name of the owner, upon which the test was made.
23. The certificate of the operator that the test was made under the specifications of the American Society for Testing Materials and that the record is a true record.
24. The signature of the operator or person responsible for the test.
25. The serial number of the test.

In the event of more than one copy of the record of any test being required, they may be furnished on separate sheets, and marked duplicates, but the original record shall always be preserved intact and complete.

For the convenience of the public, the accompanying blank form, which provides space for the necessary data, is furnished and its use recommended.

Serial No.....

REPORT OF STANDARD RATTLER TEST OF PAVING BRICK.

IDENTIFICATION DATA.

Name of the firm furnishing sample.....
Name of the firm manufacturing sample.....
Street or job which sample represents.....
Brands or marks on the brick.....
Quantity furnished..... Drying treatment.....
Date received..... Date tested.....
Length..... Breadth..... Thickness.....

STANDARDIZATION DATA.

Weight of Charge. (After Standardization.)	Condition of Locknuts on Staves.	Condition of Scales.	Number and Position of Fresh Stave Liners.	Repairs. (Note any repairs affecting the condition of the barrel.)
10 Large spheres.....				
Small spheres.....				
Total.....				

Number of charges tested since last inspection.....

RUNNING DATA.

	Time Readings.			Revolution Counter Readings.	Running Notes, Stops, etc.
	Hours.	Minutes.	Seconds.		
Beginning of Test.....					
Final Reading.....					

WEIGHTS AND CALCULATIONS.

		Percentage Loss. (Note.—The Calculation Must Appear.)
Initial Weight of Ten Bricks.....		
Final Weight of Same.....		
Loss of Weight.....		

Number of broken bricks and remarks on same.....

I certify that the foregoing test was made under the specifications of the American Society for Testing Materials, and is a true record.

(Signature of Tester).....

Date.....

Location of Laboratory.....

ACCEPTANCE AND REJECTION OF MATERIAL.

15. *Basis of Acceptance or Rejection.*—Paving bricks shall not be judged for acceptance or rejection by the results of individual tests, but by the average of no less than five tests. Where a lot of bricks fail to meet the required average, it shall be optional with the buyer whether the bricks shall be definitely rejected or whether they may be regraded and a portion selected for further test as provided in Section 16.

16. *Range of Fluctuation.*—Some fluctuation in the results of the rattler test, both on account of variations in the bricks and in the machine used in testing, are unavoidable, and a reasonable allowance for such fluctuations should be made, wherever the standard may be fixed.

In any lot of paving brick, if the loss on a test computed upon its initial weight exceeds the standard loss by more than two per cent, then the portion of the lot represented by that test shall at once be resampled and three more tests executed upon it, and if any of these three tests shall again exceed by more than two per cent the required standard, then that portion of the lot shall be rejected.

If, in any lot of brick, two or more tests exceed the permissible maximum, then the buyer may at his option reject the entire lot, even though the average of all the tests executed may be within the required limits.

17. *Fixing of Standards.*—The percentage of loss which may be taken as the standard, will not be fixed in these specifications, and shall remain within the province of the contracting parties. For the information of the public, the following scale of average losses is given, representing what may be expected of tests executed under the foregoing specifications:

	General Average Loss, per cent	Maximum Permissible Loss, per cent
For bricks suitable for heavy traffic.....	22	24
For bricks suitable for medium traffic.....	24	26
For bricks suitable for light traffic.....	26	28

Which of these grades should be specified in any given district and for any given purpose is a matter wholly within the province of the buyer, and should be governed by the kind and amount of traffic to be carried, and the quality of paving bricks available.

18. *Culling and Retesting.*—Where, under Sections 15 and 16, a lot or portion of a lot of bricks is rejected, either by reason of failure to show a low enough average test or because of tests above the permissible maximum, the buyer may at his option permit the seller to regrade the rejected brick, separating out that portion which he considers at fault and retaining that which he considers good. When the regrading is complete, the good portion shall be then resampled and retested, under the original conditions, and if it fails again either in average or in permissible maximum, then the buyer may definitely and finally reject the entire lot or portion under test.

19. *Payment of Cost of Testing.*—Unless otherwise specified, the cost of testing the material as delivered or prepared for delivery, up to the prescribed number of tests for valid acceptance or rejection of the lot, shall be paid by the buyer. (See also Section 23.) The cost of testing extra samples made necessary by the failure of the whole lot or any portion of it, shall be paid by the seller, whether the material is finally accepted or rejected.

II. VISUAL INSPECTION.

It shall be the right of the buyer to inspect the bricks, subsequent to their delivery at the place of use, and prior to or during laying, to cull out and reject upon the following grounds:

20. All bricks which are broken in two or chipped in such a manner that neither wearing surface remains substantially intact, or that the lower or bearing surface is reduced in area by more than one-fifth. Where bricks are rejected upon this ground, it shall be the duty of the purchaser to use them so far as practicable in obtaining the necessary half-bricks for breaking courses and making closures, instead of breaking otherwise whole and sound bricks for this purpose.

21. All bricks which are cracked in such a degree as to produce defects such as are defined in Section 20, either from shocks received in shipment and handling, or from defective conditions of manufacture, especially in drying, burning or cooling, unless such cracks are plainly superficial and not such as to perceptibly weaken the resistance of the brick to its conditions of use.

22. All bricks which are so off-size, or so misshapen, bent, twisted or kiln-marked, that they will not form a proper surface as defined by the paving specifications, or align with other

bricks without making joints other than those permitted in the paving specifications.

23. All bricks which are obviously too soft and too poorly vitrified to endure street wear. When any disagreement arises between buyer and seller under this item, it shall be the right of the buyer to make two or more rattler tests of the brick which he wishes to exclude, as provided in Section 2, and if in either or both tests, the bricks fall beyond the maximum rattler losses permitted under the specifications, then all bricks having the same objectionable appearance may be excluded, and the seller shall pay for the cost of the test. But if under such procedure, the bricks which have been tested as objectionable, shall pass the rattler test, both tests falling within the permitted maximum, then the buyer cannot exclude the class of material represented by this test and he shall pay for the cost of the test.

24. All bricks which differ so markedly in color from the type or average of the shipment, as to make the resultant pavement checkered or disagreeably mottled in appearance. This Section shall not be held to apply to the normal variations in color which may occur in the product of one plant among bricks which will meet the rattler test as referred to in Sections 15, 16, and 17, but shall apply only to differences of color which imply differences in the material of which the bricks are made, or extreme differences in manufacture.

APPENDIX

The following are recommended sizes of paving brick. These sizes and varieties are recognized by the Permanent Committee on Simplification of Varieties of Paving Brick of the U. S. Department of Commerce, as follows:

	DEPTH, IN.	WIDTH, IN.	LENGTH, IN.
Plain Wire-Cut (vertical fiber lugless as usually laid)	2½	4	8½
	3	4	8½
	3½	4	8½
Wire-Cut Lug (Dunn)	4	3½	8½
Repressed Lug	4	3½	8½

NOTE.—The above sizes and varieties are those recognized by the Permanent Committee, for the year 1927, and are subject to change from year to year.

STANDARD SPECIFICATIONS
FOR
CLAY SEWER BRICK[1]

Serial Designation: C 32 – 24

These specifications are issued under the fixed designation C 32; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED, 1923; REVISED, 1924.

Scope.

1. These specifications cover four classes of clay bricks intended to be used in drainage structures for the conveyance of sewage, industrial waste and storm water, as follows, the purposes for which they are intended to be especially suitable being indicated:

Class A Vitrified Brick, for the interior of structures where resistance to the abrasive action of sewage having velocities up to 18 ft. per sec. is required.

Class B Vitrified Brick, for the interior of structures where resistance to the abrasive action of sewage having velocities up to 12 ft. per sec. is required.

Hard Brick, for structures requiring imperviousness to internal and external pressures and resistance to the abrasive action of sewage having velocities up to 8 ft. per sec.

Medium Brick, for storm water conduits, where a high degree of imperviousness is not required and where abrasive action is due to sewage having low velocities only.

For definitions of the above classes, see Table I.

Basis of Purchase.

2. The consumer or purchaser shall specify the class or classes of brick to be supplied.

Acceptance.

3. (*a*) The acceptability of brick shall be determined (*1*) by the results of the chemical and physical tests hereinafter specified, and (*2*) by visual inspection, to determine whether the bricks comply with the specifications as to dimensions, shape and freedom from visible defects.

(*b*) The acceptance of brick as satisfactorily meeting one of these two general requirements shall not be construed as in any way waiving the other.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-3 on Brick.

MATERIALS

Materials. 4. (*a*) Clay bricks shall be manufactured from surface clay, fire clay or shale or a combination of these materials.

(*b*) *Surface Clay* is an unconsolidated, unstratified clay,[1] occurring on the surface.

(*c*) *Fire Clay* is a sedimentary clay[1] of low flux content.

NOTE.—It is usually associated with coal measures.

(*d*) *Shale* is a thinly stratified, consolidated sedimentary clay[1] with well-marked cleavage parallel to the bedding.

(*e*) The material shall possess such physical and chemical properties that when molded into bricks and subjected to a suitable temperature the product will be strong, durable and serviceable, free from objectionable defects and in compliance with these specifications and tests.

SIZE

Standard Sizes. 5. Sewer brick shall be not less than $2\frac{1}{4}$ by $3\frac{3}{4}$ by 8 in. nor more than $2\frac{1}{2}$ by 4 by $8\frac{1}{2}$ in., except that No. 1 vitrified brick may be 3 by 4 by $8\frac{1}{2}$ in. but not more than $3\frac{1}{2}$ by 4 by $8\frac{1}{2}$ in.

SAMPLING

6. For the purpose of tests, bricks shall be selected by a competent person appointed by the purchaser, the point or points of selection to be designated when the order is placed. The manufacturer or seller shall furnish specimens for tests without separate charge. All bricks shall be carefully examined and their condition noted before being subjected to any test. No sample which fails to meet the requirements of the visual inspection as given below shall be subjected to test. For the purpose of the tests not less than 15 bricks shall be required for each delivery; they shall be dried to constant weight in a suitable oven at a temperature of from 225 to 250° F. (107 to 121° C.).

CHEMICAL TESTS AND REQUIREMENTS

Chemical Tests. 7. The consumer or purchaser may prescribe in advance special chemical requirements in cases where sewage, industrial wastes or ground waters have a marked acid or alkaline character or are of abnormally high temperatures. He may make use of chemical analyses of the brick or brick materials to detect deleterious materials

[1] The definitions for the terms "surface clay," "fire clay," and "shale" are based upon the following definition for the term "clay":

Clay.—An earthy or stony mineral aggregate consisting essentially of hydrous silicates of alumina, plastic when sufficiently pulverized and wetted, rigid when dry, and vitreous when burned at a sufficiently high temperature.

such as caustic lime, iron pyrites or any other materials which are known to cause slaking or disintegration. Such deleterious matter contained in the brick material shall be cause for rejection.

PHYSICAL TESTS

Physical Tests.

8. (*a*) The brick shall be subjected to absorption, compression and transverse tests and shall meet the requirements specified in Table I for such tests.

(*b*) The standing of any set of bricks shall be determined by that one of the three requirements in which it is the lowest.

Absorption.

9. (*a*) At least five bricks after being dried to constant weight shall be completely submerged in water at a temperature of between 60 and 80° F. (15 and 27° C.). The water shall be heated to boiling within one hour, boiled continuously for five hours and then allowed to cool to a temperature between 60 and 80° F. (15 and 27° C.). The brick shall then be removed, the surface water wiped off with a damp cloth and the bricks quickly weighed.

(*b*) The percentage of absorption shall be calculated on the dry weight from the formula:

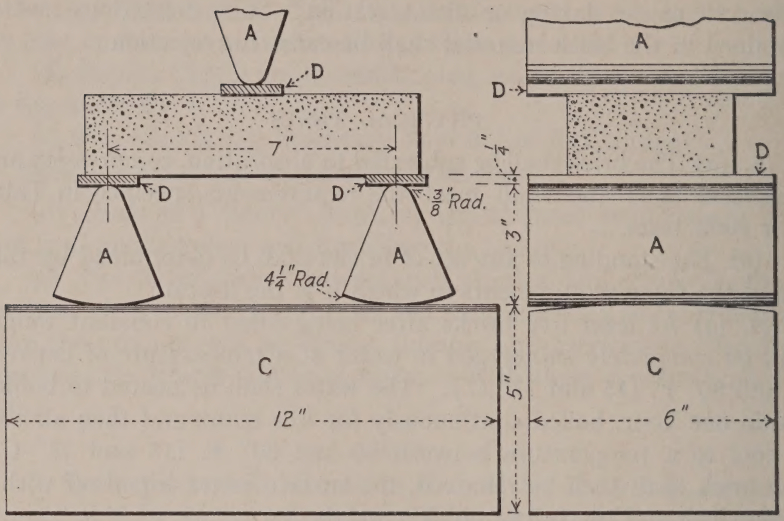
$$\text{Percentage of Absorption} = \frac{100\ (b-a)}{a}$$

where a = weight of dry brick and b = weight of saturated brick.

Compression Test.

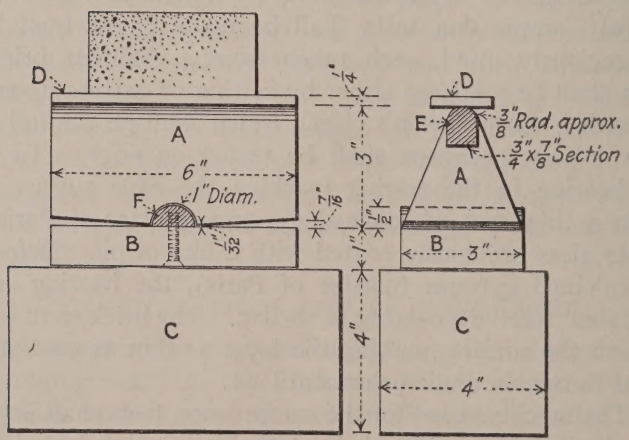
10. (*a*) Compression tests shall be made on at least five half bricks, previously dried, each taken from a different brick. The half brick shall be prepared either by sawing or cutting upon a yielding bed with a sharp mason's chisel, which shall be the full width of the brick. The specimens shall be tested on edge. To secure a uniform bearing in the testing machine the edge surface shall be bedded in a thin coat of calcined gypsum (plaster of Paris) spread upon plate glass previously coated with a film of oil. Before applying the calcined gypsum (plaster of Paris), the bearing surface of the brick shall receive a coating of shellac. The brick shall be pressed firmly upon the surface, making the layer as thin as possible and be permitted to remain undisturbed until set.

(*b*) The machine used for the compression tests shall be equipped with a spherical bearing block kept thoroughly lubricated to insure accurate adjustment, which should be made by hand under a small initial load. The radius of the spherical bearing surface should be such that the center of the sphere falls in the adjacent end surface of the specimen. During the test the beam of the testing machine shall be kept constantly in a floating position.



A = Cast-Iron Wedges; C = Soft Wood Block; D = Cold-Rolled Steel Plates.

FIG. 1.



A, B = Cast-Iron Wedges; C = Soft Wood Block;
D, E, F = Cold-Rolled Steel.

FIG. 2.

(*c*) The breaking load shall be divided by the area in compression and the results reported in pounds per square inch.

Transverse Test.

11. (*a*) At least five bricks, previously dried, shall be tested, laid flat-wise on knife edges with a span of 7 in. and with the load applied at midspan. Steel bearing plates, about $\frac{1}{4}$ in. thick by $1\frac{1}{2}$ in. wide, may be placed between the knife edges and the brick. The use of a wooden base block, slightly rounded transversely across the top, upon which to rest the lower knife edges (see Fig. 1), or the form of lower knife edge shown in Fig. 2, is recommended. If the knife edges shown in Fig. 2 are used, they should rest upon smooth plane blocks of wood at least 2 in. thick.

(*b*) The modulus of rupture shall be calculated in pounds per square inch by the following formula:

$$R = \frac{3\ Wl}{2\ bd^2}$$

in which l equals the distance between supports in inches, b equals the breadth and d equals depth of the brick in inches, and W equals the load in pounds at which the brick failed. The measurement d is taken at the fractured surface after the specimen is broken.

TABLE I.—PHYSICAL TEST REQUIREMENTS FOR DIFFERENT CLASSES OF BRICK.

Class.	Absorption Limits, per cent.		Compressive Strength (on edge), lb. per sq. in.		Modulus of Rupture, lb. per sq. in.	
	Mean of 5 Tests.	Individual Maximum.	Mean of 5 Tests.	Individual Minimum.	Mean of 5 Tests.	Individual Minimum.
Class A Vitrified.	3 or less	....	5000 or over	....	1200 or over	...
Class B Vitrified.	5 or less	6.0	5000 "	4000	1200 "	800
Hard.....	5 to 10	12.0	3500 "	2500	600 "	400
Medium.....	10 to 15	17.0	2000 "	1500	450 "	300

VISUAL INSPECTION

Visual Inspection.

12. (*a*) All brick shall be given a thorough visual inspection at the sewer trench by a competent inspector employed by the purchaser to determine whether the brick, independently of the chemical and physical requirements, comply with the specifications of general properties as follows:

(*b*) Bricks shall be of rectangular cross-section, full size and of uniform dimensions throughout. Recessed bricks shall not be accepted.

(*c*) Bricks shall have straight, parallel edges and square corners. The ends and at least one edge shall have a smooth plane surface; kiln

marks not exceeding $\frac{1}{16}$ in. in depth shall be permitted on the opposite edge.

(*d*) Bricks shall be thoroughly and uniformly burned and when broken shall show a fracture of uniform and compact texture.

(*e*) The color of the bricks shall be considered to indicate their burning, providing the meaning of the variation in color be first determined from an investigation of the materials and the process of manufacture.

(*f*) Bricks shall be free from checks or cracks extending into the body of the brick in a manner that would affect their serviceability or strength. Bricks shall be free from stones, pebbles, and visible grains or masses of lime.

(*g*) Bricks when struck together or when struck with a trowel shall give a clear ringing sound.

STANDARD METHOD OF TEST
FOR
REFRACTORY MATERIALS UNDER LOAD AT HIGH TEMPERATURES [1]

Serial Designation: C 16 - 20

This method is issued under the fixed designation C 16; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1917; ADOPTED IN AMENDED FORM, 1920.

Scope.

1. The object of this test is to determine the resistance of the specimen to deformation at a specified temperature for a specified time, when subjected to a compressive load of 25 lb. per sq. in. (1.765 kg. per sq. cm.).

Apparatus.

2 The apparatus consists essentially of a furnace and loading device It shall be constructed in accordance with Figs. 1 and 2.

(*a*) The furnace shall be cylindrical in form, 18 in. (457 mm.) in internal diameter, as shown in Fig. 1 (Plate II).

(*b*) The heating shall be done with gaseous or oil fuel and compressed air, using not less than two burners located tangentially and so arranged that no flame can impinge upon the test specimen. The burners shall be such as will insure a uniform temperature in all parts of the furnace and be under complete control.

(*c*) The method of loading shown in Fig. 1 shall be used, and the details shall be such as will insure accuracy in the applied load and freedom from eccentric loading, both in the original application and during the testing. It is advantageous to make the cross-beams as light as possible, so that the greater portion of the load may be concentrated in the weights.

(*d*) The temperature may be measured either with a calibrated platinum-rhodium thermocouple, encased in a double protecting tube with the junction not more than 1 in. (25 mm.) from the side or edge of the specimen and approximately opposite the center; or with some form of optical pyrometer that has

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

been calibrated against a thermocouple in the furnace. If the thermocouple is used, the cold-end temperature should be kept constant in melted ice. A recording form of indicator is recommended where possible.

Test Specimen.

3. The test specimen shall consist, whenever possible, of a standard 9-in. brick placed vertically on end. In the case of

TABLE I.—TEMPERATURE TO BE ATTAINED AT TIME SPECIFIED.

ALL TEMPERATURES IN DEGRESS CENTIGRADE.

Time.	Silica.	Fire Clay.		
		Heavy Duty.	Moderate Duty.	Light Duty.
Hr. Min.				
0 15	40	160	160	160
30	80	280	280	280
45	140	400	400	400
1 0	200	500	500	500
15	260	620	595	570
30	290	720	685	640
45	300	815	770	700
2 0	310	900	850	755
15	320	980	920	810
30	385	1045	990	860
45	490	1100	1050	905
3 0	590	1150	1100	950
15	695	1195	1145	985
30	800	1235	1185	1020
45	900	1270	1220	1050
4 0	1000	1300	1250	1075
15	1100	1330	1275	1090
30	1200	1350	1300	1100
45	1250	1350	1300	1100
5 0	1300	1350	1300	1100
15	1350	1350	1300	1100
30	1380	1350	1300	1100
45	1410	1350	1300	1100
6 0	1440	1350 (End)	1300 (End)	1100 (End)
15	1470			
30	1500			
45	1500			
7 0	1500			
15	1500			
30	1500			
45	1500			
8 0	1500 (End)			

blocks or shapes, sections approximately 9 by $4\frac{1}{2}$ by $2\frac{1}{2}$ in. (228 by 114 by 64 mm.) shall be cut, utilizing as far as possible existing plane surfaces. The ends of the specimen shall be either ground so that they are parallel and perpendicular to the vertical axis, or if this is impossible, shall be bedded in a neutral cement, so that the specimen is perpendicular to the base of the furnace.

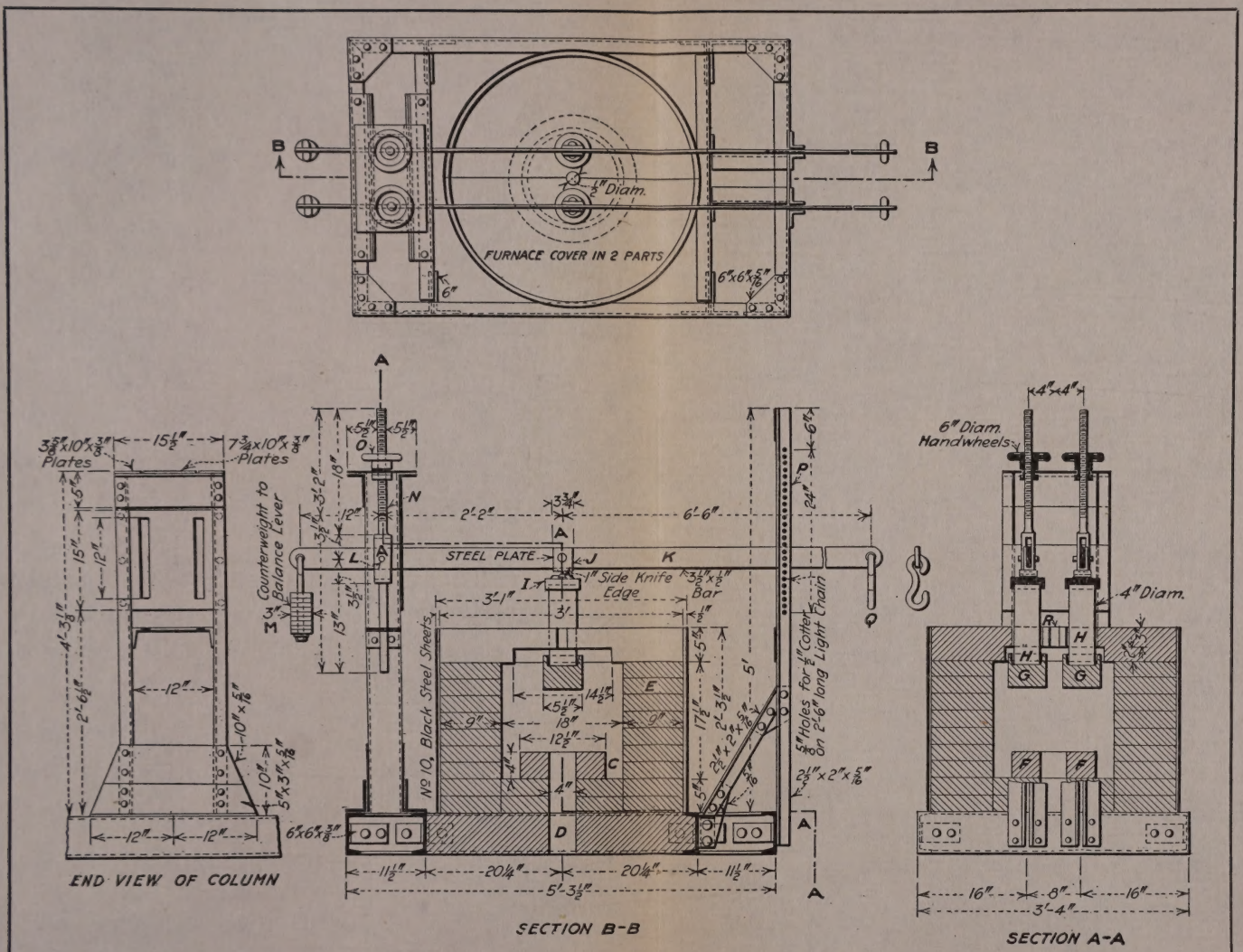
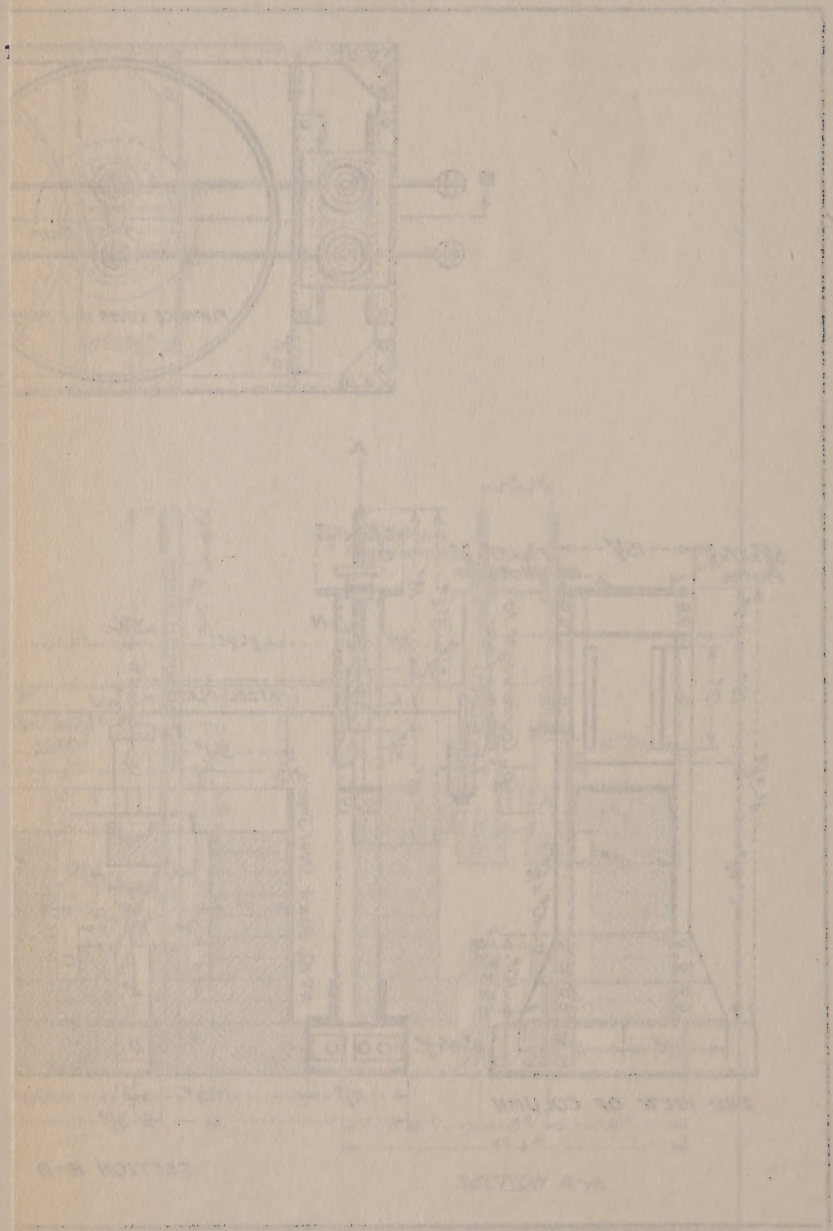


FIG. 1.—Apparatus for Testing Refractory Materials under Load at High Temperatures.
(By courtesy of *Chemical and Metallurgical Engineering*).



The test specimen shall be measured before testing, making not less than five observations in each direction to within ±0.02 in. (0.5 mm.). The average dimensions shall be reported, and the cross-section calculated.

4. The test specimen shall occupy approximately the center of the furnace and should rest on a block of some highly refractory material, having a minimum expansion or contraction. A silicon-carbide brick has been found satisfactory. At the top of the test specimen a block of similar highly refractory material should be placed, extending through the furnace top to receive the load. Starting the Test.

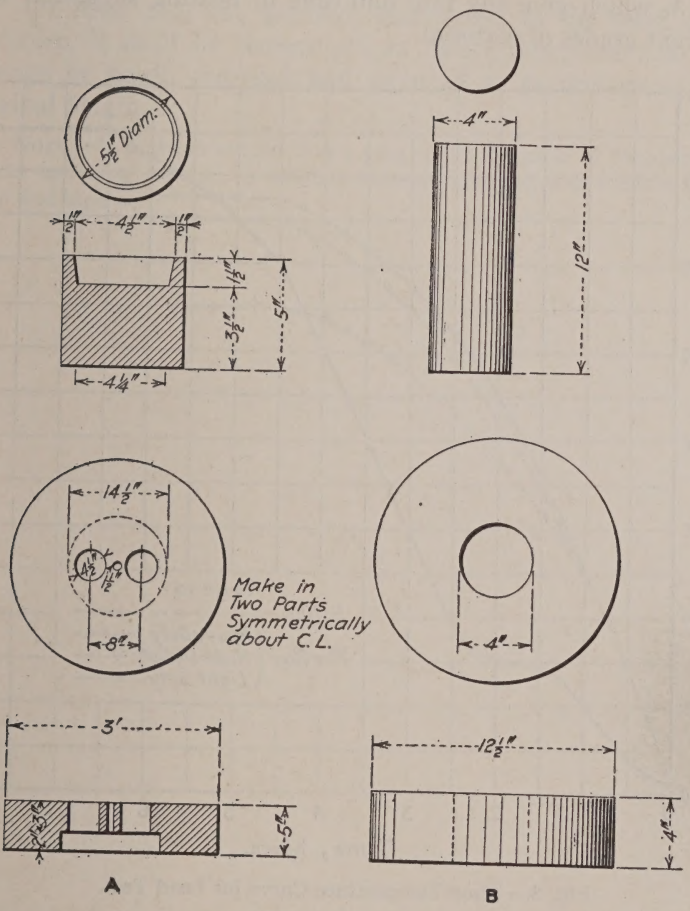


FIG. 2.—Special Shapes Required for Furnace.

(By courtesy of *Chemical and Metallurgical Engineering.*)

NOTE.—Gross errors which may more than double the contraction will result if the specimen is not set perpendicular to the base of the support or if the load is eccentrically applied.

Heating. 5. The rate of heating shall be in accordance with the requirements of Table I and the time-temperature curves of Fig. 3, which give the rate and time of heating suggested for different grades of material.

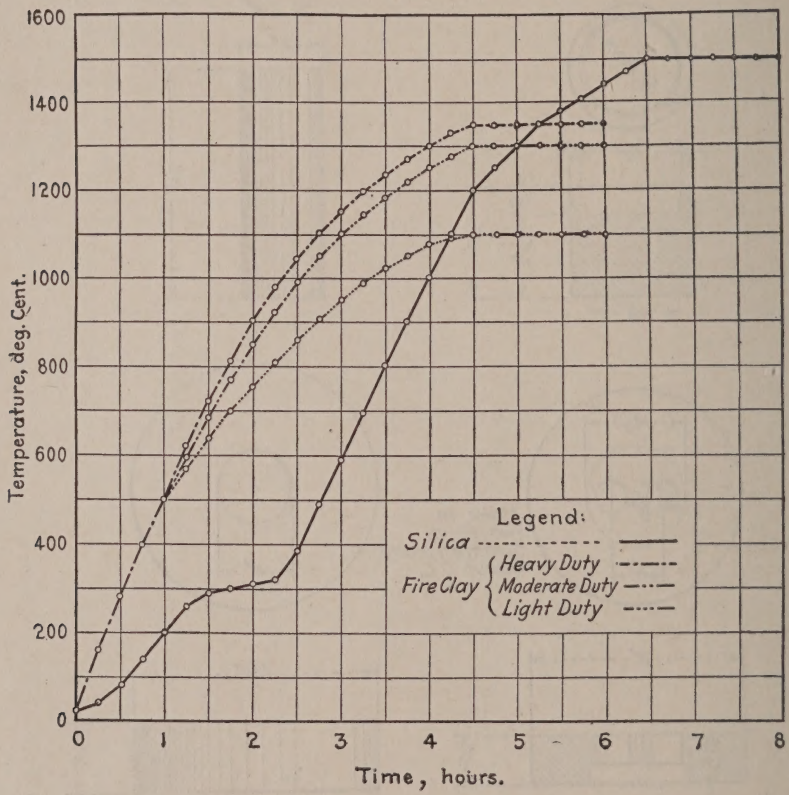


FIG. 3.—Time-Temperature Curve for Load Test.

Loading. 6. (*a*) The load is calculated from the average cross-section as determined on the untested specimen and the requirement of the test. It is recommended that for general purposes, 25 lb. per sq. in. (1.765 kg. per sq. cm.) be used.

(*b*) The additional masses required to give the desired loading should be equally distributed on each side of the beam.

Completing the Test. 7. (*a*) At the expiration of the time of heating, the supply of heat shall be stopped and the furnace allowed to cool, during

not less than 5 hours before removing the load and examining the test specimen.

NOTE.—The specimen shall be examined immediately after the heating is stopped for evidences of cracking and spalling, as such defects may develop later due to the rapid cooling of the furnace.

(*b*) After the test specimen has cooled to the room temperature, it shall be remeasured as before described, and the change in length recorded and reported as percentage of the original length.

NOTE.—It is recommended that a photograph be made of the specimen before and after testing, as yielding valuable information at a minimum of time and expense.

STANDARD METHOD OF TEST
FOR
POROSITY AND PERMANENT VOLUME CHANGES IN REFRACTORY MATERIALS[1]

Serial Designation: C 20 – 20

This method is issued under the fixed designation C 20; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1918; ADOPTED, 1920

Scope.

1. The object of this test is to determine the porosity and permanent volume changes in refractory materials when heated to series of specified temperatures.

Preparation of Test Specimens.

2. (*a*) The sample shall consist of at least seven standard-size bricks.

(*b*) Test specimens measuring $2\frac{1}{2}$ by $2\frac{1}{2}$ by $1\frac{1}{4}$ in. shall be cut so as to remove the original surfaces of the bricks; for this a "cut off" grinding wheel is recommended. There should be five test specimens for each of the seven heat treatments specified in Section 4 or 35 test specimens for each kind of brick. The test specimens shall be brushed or washed free from all adhering dust and marked serially with a refractory stain, for which 5-per-cent cobalt-kaolin mixture is suggested.

Procedure.

3. After the test specimens have been cut and cleaned, they shall be dried and the volumes and porosity of each obtained as described in Sections 5 and 6. They shall be heated as specified in Section 4, and the changes in volume and porosity determined.

Burning.

4. (*a*) Dry the test pieces prior to placing in the kiln.

(*b*) Raise the temperature as rapidly as is consistent with even heat distribution to 1200° C. From 1200° C. raise the temperature at the rate of 30° per hour, drawing samples at each 50° interval from 1200 to 1500° C.

(*c*) If it is possible and the number of brands being tested warrant, it is best that separate burns to each temperature be made and the kiln sealed and allowed to cool by radiation. In case separate burns cannot be made, the five test specimens from each temperature

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

increment should be covered with hot sand immediately on being drawn; or placed in a supplementary furnace and kept at about 500° C. until all drawings are completed, and then cooled with the furnace sealed to cool wholly by radiation.

Method of Obtaining Data.

5. (*a*) The test specimens shall be cleaned from adhering or loosely attached pieces and particles, care being taken not to alter the exterior volume as originally prepared for this test.

(*b*) The test specimens shall be heated if necessary to 110° C. to remove moisture, and their dry weight (*D*) obtained to 0.10 g.

(*c*) The test specimens shall be placed in kerosine of known density (δ) under a vacuum of 24 in. for 4 hours at 25° C. and cooled to room temperature while yet immersed.

(*d*) When cool, each test specimen shall be weighed suspended in kerosine at 25° C. to determine its *Suspended Weight* (*S*), in grams.

(*e*) The *Saturated Weight* (*W*), in grams, of each test specimen shall be obtained immediately after obtaining the suspended weight, by drying lightly with a kerosine-moistened towel to remove the excess kerosine and then weighing in air.

(*f*) The *Exterior Volume* (*V*), in cubic centimeters, of each test specimen is obtained by subtracting the suspended weight (*S*) from the saturated weight (*W*), and dividing by the density (δ) of the kerosine. Thus:

$$V = \frac{W - S}{\delta} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

(*g*) The *Actual Volume of Open Pores* (V_1) in cubic centimeters, is obtained by subtracting the dry weight (*D*) from the saturated weight (*W*), and dividing by the density (δ) of the kerosine. Thus:

$$V_1 = \frac{W - D}{\delta} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (2)$$

(*h*) The *Apparent Specific Gravity* (T_1) of that portion of the test specimen which is impervious to liquid is obtained by dividing the dry weight by the difference between the dry and suspended weights, and multiplying by the density of the kerosine. Thus:

$$T_1 = \frac{D}{D - S}\,\delta \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (3)$$

(*i*) The *True Specific Gravity* (*T*) of the wholly solid or burned clay portion is obtained by crushing a portion of the dried test specimen to powder passing a No. 120 sieve and determining the displacement at 25° C. under 24 in. vacuum, of a 20-g. sample in a 50-cc. straight-

wall pyknometer using kerosine, and correcting for density of the kerosine.

(*j*) The *Volume of Sealed Pores* (V_2) in cubic centimeters, is obtained by subtracting the quotient of dry weight (D) divided by true specific gravity (T) from the volume of the impervious portion of the test specimen; or:

$$V_2 = \frac{(D - S)}{\delta} - \frac{D}{T} \quad \ldots\ldots\ldots\ldots\ldots\ldots (4)$$

(*k*) The *Volume Shrinkage* is obtained by subtracting the volumes, that is, the values of $\frac{W - S}{\delta}$, before and after the heat treatment.

Basis of Reference for the Data.

6. To show progressive changes in the several volumes, refer all volumes back to the original exterior volume of the test specimen as 100. This is done by multiplying all volumes by the factor $100/V$, in which V is the exterior volume of the test specimen prior to the subjection to heat treatment.

The volume data should be determined for each test specimen and multiplied by the above factor to reduce all volumes for each test specimen to terms of 100 original exterior volumes of that test specimen before the average of the five for each heat treatment is calculated.

STANDARD METHOD OF TEST
FOR
SOFTENING POINT OF FIRE-CLAY BRICK[1]

Serial Designation: C 24 – 20

This method is issued under the fixed designation C 24; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1919; ADOPTED, 1920.

Scope.

1. The object of this test is to determine the softening point of fire-clay brick, by comparison of test cones with standard Orton pyrometric cones heated in a suitable furnace.

Preparation of Sample.

2. A 1-kg. (2-lb.) sample shall be taken by chipping off approximately equal portions from the corners of the brick. These fragments shall be reduced in size by means of rolls or a jaw crusher adjusted to pass a lump 6 mm. ($\frac{1}{4}$ in.) in diameter. They shall be mixed thoroughly, and the amount of material reduced to about 250 g. (5 lb.) by quartering. A magnet shall be repeatedly passed through the crushed material until all particles of metallic iron are removed. This portion shall be ground in a porcelain or agate mortar to pass a No. 60 sieve of the United States Standard Sieve Series.[2] In order to avoid excessive reduction of the fines, they shall be removed frequently during the process of reduction by throwing the sample on the sieve and continuing the grinding of the coarser particles until all the sample will pass through the sieve.

Preparation of Test Cones.

3. (*a*) The sample thus prepared shall be thoroughly mixed and after the addition of sufficient dextrine or glue and water, shall be formed into test cones in a metal mold in the shape of tetrahedrons measuring 5 mm. ($\frac{3}{16}$ in.) on the sides at the base and 25 mm. (1 in.) high.

(*b*) When dry the test cones may be subjected to a preliminary burn at a temperature not exceeding 1300° C. (2372° F.) for the purpose of sintering them into a firm condition to permit handling.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] For detailed specifications for this sieve, see the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

Mounting. 4. The test cones shall be mounted on plaques of refractory material of such a composition as will not affect the fusibility of the cones.[1] They shall be mounted with the base embedded approximately 1 mm. (0.04 in.) in the plaque and the face of one side inclined at an angle of 75 deg. with the horizontal. The arrangement with respect to the Orton cones shall be sub-

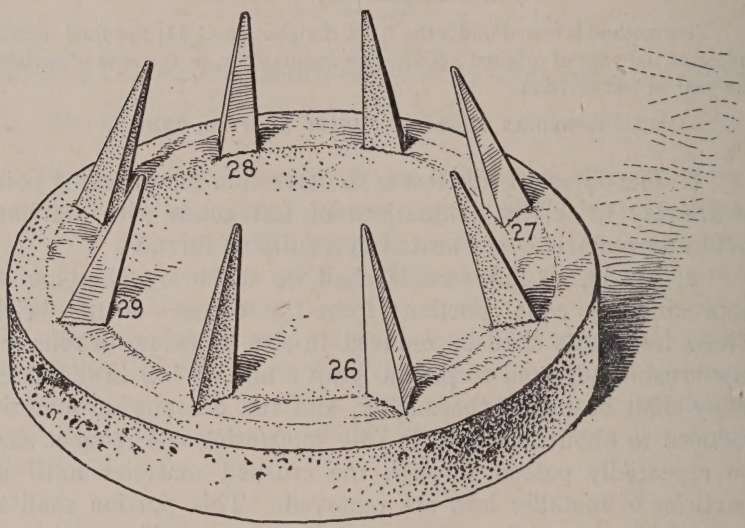


FIG. 1.

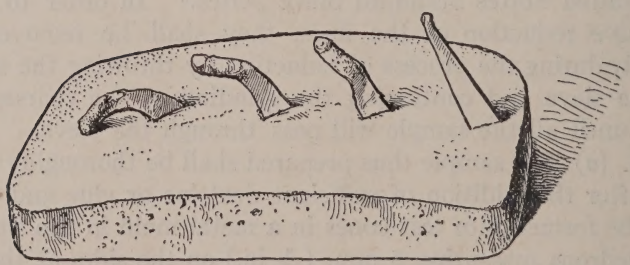


FIG. 2.

stantially as shown in Fig. 1, that is, alternating with the Orton cones in such a way that Orton cones of successive numbers will be placed opposite each other. The plaque may be any convenient size and shape and may be biscuited before using, if desired.

[1] A mixture of equal parts of a highly refractory clay such as a good grade of china clay, and fused alumina which will pass a No. 100 sieve has been found satisfactory.

5. (*a*) The heating shall be done in a suitable furnace at a rate not greater than 15° C. (27° F.) per minute, nor less than 10° C. (18° F.) per minute after cone No. 1 is reached, or as nearly within these limits as possible. Heating.

(*b*) That type of furnace in which a neutral or oxidizing atmosphere may be maintained is to be preferred. Excessive reducing conditions should be avoided. Care should be taken that the flame does not strike directly against the cone or cone plaque. The furnace should be tested at intervals for the determination of the uniformity of the distribution of the heat.

6. (*a*) The softening of the cone will be indicated by the top bending over and assuming the position shown in Fig. 2. The bloating, squatting or unequal fusion of small constituent particles should always be reported. The softening point shall be reported in terms of Orten cones and shall be that cone which most nearly corresponds in time of softening with the test cone. If the test cone softens later than one Orton cone but earlier than the next Orton cone and approximately midway between, the softening point shall be reported thus: Cone No. 31–32. Softening Point.

(*b*) If the test cone starts bending at an early cone but is not down until a later cone, the fact should be reported.

STANDARD METHODS
OF
ULTIMATE CHEMICAL ANALYSIS OF REFRACTORY MATERIALS, INCLUDING CHROME ORES AND CHROME BRICK[1]

Serial Designation: C 18 – 21

These methods are issued under the fixed designation C 18; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1917; ADOPTED IN AMENDED FORM, 1920; REVISED, 1921.

Prefatory Note.—It will be understood that the making of a complete silicate analysis is a difficult procedure requiring a wide knowledge of the chemistry involved in the operations, and a thorough training in carrying out the work. A skilled analyst of good training is therefore required to do the work. The descriptions here given cover the vital points of procedure, but frequent reference in regard to the details of the various manipulations must be made to *Bulletin No. 422*, United States Geological Survey on "Analysis of Silicate and Carbonate Rocks," by W. F. Hillebrand; also, to "Treatise on the Ceramic Industries," Vol. 1 (1913), by J. W. Mellor; and to similar publications.

GENERAL REFRACTORIES

GENERAL CONSIDERATIONS

Grinding of Sample.

1. The sample shall be crushed in a hardened tool-steel mortar, using a pestle of the same sort. Fine grinding shall be done in an agate mortar, either by hand, or by a mechanical sample grinder of the McKenna, Carling or similar type, so constructed as to prevent the introduction of impurity.

Statement of Analysis.

2. Moisture shall be determined in the sample in its ordinary air-dried condition, and all other percentage compositions shall be calculated to a moisture-free basis. Whenever a sample is weighed out for any determination, a moisture determination shall also be made. If preferred, the sample may be dried in a weighing bottle, from which the required samples shall be weighed out.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

3. In all cases, check determinations shall be made, and the results shall be redetermined if satisfactory checks are not obtained. It shall be considered satisfactory if the differences between check determinations do not exceed the following limits: Checking Results.

For silica or other constituent amounting to 30 per cent or over	0.3 per cent
For alumina or other constituent amounting to 10–30 per cent	0.2 per cent
For any other constituent amounting to under 10 per cent	0.1 per cent

These figures are stated in terms of the whole sample as 100 per cent.

SOLUTIONS REQUIRED

4. The acids referred to as concentrated shall be of approximately the following specific gravities: Concentrated Acids.

Hydrochloric Acid (HCl)	1.19
Sulfuric Acid (H_2SO_4)	1.84
Nitric Acid (HNO_3)	1.42

5. Dissolve 1.5 g. of ammonium carbonate in 50 cc. of cold water. Ammonium Carbonate.

6. Dissolve 107 g. of NH_4Cl in 1000 cc. of warm water. Ammonium Chloride.

7. Neutralize 20 cc. of concentrated HNO_3 with NH_4OH and dilute to 1000 cc. Test with litmus; the solution shall not be acid. Ammonium Nitrate.

8. Dissolve 1 g. of ammonium oxalate in 50 cc. of water, heating gently. Ammonium Oxalate.

9. The strength of the final standard titania solution shall be 1 cc. = 0.0001 g. TiO_2. To prepare the solution weigh out in a platinum crucible an amount of potassium titanium fluoride (K_2TiF_6) sufficient to make from ½ to 1 liter of "stock solution" in which 1 cc. = 0.001 g. TiO_2. Evaporate several times with H_2SO_4 without taking to dryness, thus driving out all fluorine. Take up the residue with water containing enough H_2SO_4 to make at least 5 per cent of the solution, when finally diluted to the standard solution strength. To standardize the stock solution take out two 50-cc. portions, dilute, boil and precipitate with ammonia. Filter, wash with hot water until free from alkali, ignite, blast and weigh. The duplicate should check very closely. From the weight of titania thus determined calculate Standard-Titania Solution.

the strength of the stock solution. The standard solution to be used is obtained from the stock solution by diluting until 1 cc. = 0.0001 g. (TiO_2).

Standard Potassium-Permanganate Solution.

10. Dissolve about 0.4 g. (a little less) of $KMnO_4$ in 1 liter of water and check against an iron solution of known purity and strength. Dilute the solution until 1 cc. = 0.001 g. Fe_2O_3

The ordinary chemical iron wire is not sufficiently pure for this use. If preferred the solution may be standardized against sodium oxalate, a pure form of which is prepared especially for such work by the U. S. Bureau of Standards, Washington, D. C.

METHODS

MOISTURE.

Moisture.

11. To determine moisture, heat approximately 1 g. of the sample at a temperature not under 105 nor over 110° C., to constant weight.

LOSS ON IGNITION.

Ignition Loss.

12. To determine loss on ignition, heat about 1 g. of the sample over a blast lamp or in an electric furnace at 900 to 1000° C., to constant weight. Ignition loss may be determined in the sample from which the moisture has been removed. The percentage of ignition loss is calculated to a moisture-free basis.

SILICA.

Silica.

13. To determine silica, weigh out approximately 0.5 g. of the sample and mix with 5 g. of Na_2CO_3. Put a little Na_2CO_3 in the bottom of the crucible before introducing the mixture, and then cover the mixture with a little more Na_2CO_3. Fuse over a Meker burner or blast lamp until complete solution is obtained. Cool the fusion and as it solidifies, rotate the crucible to spread the mass up the side walls. Dissolve the fusion in about 100 cc. of water in a platinum or porcelain evaporating dish. Add about 20 cc. of concentrated HCl, introducing it slowly by means of a pipette, keeping the dish covered with a watch glass. Evaporate to dryness and heat until the fumes of HCl are gone. Add about 5 cc. of concentrated HCl and 30 to 40 cc. of water. Warm on a water bath for 10 to 15 minutes and break up the lumps. Decant the clear liquid onto a filter paper and collect the filtrate in a 400-cc. beaker. Add more HCl and water to the evaporating dish, warm again and decant. Repeat this a third time.

Finally transfer the contents of the dish to the filter paper.

Wash with cold water until silver nitrate shows no chlorides to be left. Transfer the filtrate to the original evaporating dish, evaporate again to dryness, take up with a little HCl and water, transfer to a new filter paper and wash as before. Transfer both silica precipitates to a platinum crucible. Ignite carefully over a Bunsen flame until the filter paper is burned off, then blast for about 30 minutes; cool and weigh. Repeat blasting for 5 minutes, weigh again and repeat until constant weight is obtained.

To the residue in the crucible add about 5 cc. of water and 5 drops of H_2SO_4. Add HF drop by drop at first, and then slowly until the crucible is almost one-half filled. Warm on a hot plate until almost dry, add 2 or 3 cc. more of HF and evaporate to dryness. Heat the crucible to bright redness and then blast for 5 minutes. Cool and weigh and repeat blasting to constant weight. The loss in weight from the original silica residue represents the actual silica content (SiO_2), except for that part of silica which is later recovered from alumina, etc. The residue from the HCl evaporation is left in the crucible and the total precipitate of alumina, etc., is added to this same crucible in which it is ignited and weighed.

ALUMINA.

Alumina.

14. Regarding the determination of alumina, reference should be made to *Scientific Paper No. 286* of the U. S. Bureau of Standards by Dr. Blum on "Determination of Alumina as Oxide."

To determine alumina, to the filtrate from the silica determination, add about 10 cc. of NH_4Cl solution and heat to boiling. Then add NH_4OH very slowly and with constant stirring until there is a slight excess. The NH_4OH must be free from CO_2. Filter hot and rapidly, and wash four times by decantation with hot solution of ammonium nitrate. Carefully wash the precipitate from the filter paper into a beaker, using hot water. Dissolve the precipitate in hot dilute HCl. Repeat the precipitation with NH_4Cl and NH_4OH. Decant four times as before, using hot ammonium nitrate. Transfer to the filter paper and wash with hot ammonium nitrate until the washings are free from chlorides when tested by silver nitrate. Evaporate the filtrate nearly to dryness, add a little NH_4OH and continue the evaporation. Keep the solution alkaline to coagulate any iron and aluminum hydroxides. Transfer the precipitate to a filter paper and wash thoroughly. Transfer the moist filter paper to the plat-

inum crucible containing the residue from the silica. Burn off the filter paper and blast the precipitate. Repeat blasting and weighing to a constant weight.

IRON OXIDE.

Iron Oxide.

15. To determine iron oxide, fuse the ignited alumina precipitate with about six times its weight of potassium pyrosulfate or potassium bisulfate. Avoid sputtering. Heat carefully to redness and continue heating until the residue is all dissolved. Cool. Dissolve in warm water and add about 10 cc. of dilute H_2SO_4 (1:5). Evaporate to a small volume. Then heat to a higher temperature until copious fumes of H_2SO_4 are evolved. Sufficient H_2SO_4 should be present to form a pasty mass when cooled. Take up with water, filter off, wash, ignite and weigh the silica as before and evaporate with HF as before. The loss in weight is extra silica to be added to the original silica determination and subtracted from the alumina. Fuse the residue with a little potassium pyrosulfate or potassium bisulfate, and add the solution of this cake to the main solution. Transfer the total filtrate to a 250-cc. graduated flask and dilute to 250 cc., mixing thoroughly. Draw off 50 cc. of this solution and reserve for titania determination. Pass the remaining 200 cc. through a Jones reductor, or otherwise reduce the solution with pure zinc and H_2SO_4. To the solution add 3 cc. of 10-per-cent $CuSO_4$ solution and titrate with standard $KMnO_4$ solution (1 cc. equivalent to 0.001 g. Fe_2O_3), until a faint pink tinge is seen. If Jones reductor or similar apparatus is used, subtract the $KMnO_4$ equivalent, which has previously been determined on blank samples run through the apparatus. From the result, calculate the iron as Fe_2O_3.

TITANIA.

Titania.

16. To determine titania, place in a small Nessler tube the 50 cc. of filtrate previously set aside for titania determination, and fill the tube up to the graduation mark. Add 1 cc. of H_2O_2 (free from fluorine) and shake well. The color of this solution is now compared in any standard color comparator with the color of a known standard solution, preferably of such strength that 1 cc. equals 0.0001 g. of titanium dioxide (TiO_2). To make the comparison, put 10 cc. of this standard solution into a second small Nessler tube and dilute with water from a burette until the color is matched. Note the amount of water added and calculate the percentage of TiO_2 in the sample.

LIME.

Lime. 17. To determine lime, evaporate the filtrate from the alumina precipitation to about 250 cc. While still boiling add about 5 cc. of acetic acid. Meanwhile, dissolve about 1 g. of oxalic acid in a little hot water and add to the solution. In about 5 minutes add a slight excess of NH_4OH and boil until precipitation is complete. Cool thoroughly and filter. Wash three times by decantation with dilute NH_4OH (1 : 10) or a 1-per-cent solution of ammonium oxalate. Dissolve the precipitate by adding about 50 cc. of dilute HNO_3 (1 : 5). Again add a slight excess of NH_4OH and a few drops of oxalic-acid solution. Boil well and let stand a couple of hours to cool. Filter and wash as before. Transfer the precipitate to a platinum crucible, carefully burn off the paper and ignite over a blast lamp for about 10 minutes. Cool and weigh quickly. Repeat the blasting to constant weight. The increase in weight of crucible is calcium oxide.

MAGNESIA.

Magnesia. 18. To determine magnesia, add to the filtrate from lime precipitation about 2 g. of sodium-ammonium-phosphate dissolved in 15 cc. of water. Stir vigorously and while stirring add drop by drop about one-third of the volume of the solution of NH_4OH. Let stand 12 to 24 hours. Filter and wash the precipitate with dilute NH_4OH (1 : 10). Discard the filtrate. Wash the precipitate until the washings show no discoloration when tested with silver nitrate. Redissolve the precipitate in warm dilute HNO_3 (1 : 5), using about 50 cc., and collect in a beaker. Precipitate and wash as before. Collect the precipitate on a Gooch asbestos crucible. Dry slowly and then heat over a Meker burner for about 10 minutes; cool and weigh. Re-ignite to constant weight.

The $Mg_2P_2O_7$ is never pure, being contaminated by small amounts of calcium, barium, aluminum, iron, manganese, etc., but in the analysis of refractories this error is negligible, and correction for these impurities is not required unless previously agreed upon.

ALKALIES.

Alkalies. 19. To determine alkalies, the J. Lawrence Smith method shall be used. Weigh out about 0.5 g. of the sample and mix

well with 0.5 g. of NH_4Cl and 3 g. of $CaCO_3$. Transfer to a platinum crucible, placing about 0.5 g. of $CaCO_3$ in the bottom of the crucible and a similar amount over the top of the mixture. Heat gently over a low flame for about 15 minutes to volatilize NH_4Cl. Then raise the temperature until the lower three-quarters of the crucible is dull red. Hold this temperature for about one hour. Cool, take up with about 50 cc. of water and heat over a water bath, adding water to replace that lost by evaporation. Break up any lumps with a small pestle. Decant the clear liquid through a filter paper and wash four times by decantation. Then transfer the residue to the filter. Wash until silver nitrate shows only a very faint turbidity. To the filtrate add NH_4OH and ammonium carbonate and heat to boiling. Filter and again digest the precipitate with NH_4OH and ammonium carbonate. Filter and allow the filtrate to collect with the previous filtrate, then evaporate to dryness in a platinum or porcelain dish. Remove ammonium salts by gentle ignition in a moving flame. Treat with water and remove the last trace of lime by adding ammonium oxalate to the boiling solution and let stand over night. Filter, evaporate to dryness, ignite gently and allow to cool. Then moisten the residue with HCl, evaporate again to dryness, ignite gently and weigh. Dissolve in water. Ignite and weigh any insoluble residue, deducting this from the alkali chlorides.

Dilute the solution of the mixed chlorides and add hydrochloroplatinic acid in amount equal to 0.3 cc. more than would be necessary if all chlorides were present as NaCl. Evaporate to a syrupy consistency. Cool and treat with a few cubic centimeters of 80-per-cent ethyl alcohol. Stir and decant through a weighed Gooch crucible. Treat again with 80-per-cent alcohol and decant. Repeat washing and decantation until the alcohol leaving the Gooch crucible is colorless and the precipitate appears golden yellow, not orange. Transfer the precipitate to the Gooch crucible, wash with 80-per-cent alcohol about six times and dry at 130° C. to constant weight. This residue is potassium chloroplatinate.

The oxides are calculated as follows:

Weight of potassium chloroplatinate $\times 0.3068 = KCl$.
Weight of potassium chloroplatinate $\times 0.1941 = K_2O$.
Total chlorides $- KCl = NaCl$.
$NaCl \times 0.5303 = Na_2O$.

CHROME ORES AND CHROME BRICK

Sampling. 20. The sample shall be secured after the manner of any of the approved methods of sampling. If from ores or chrome-bearing refractories, it shall not be less than 10 lb., in small nut-size particles when sent to the laboratory. If from chrome brick, the sample shall consist of a small portion from the interior of from 15 to 25 bricks. This 10-lb. sample shall be crushed to pass a No. 20 sieve[1] and quartered until a sample of 100 g. is secured, which shall be ground to pass a No. 100 sieve.[1] From this portion, a smaller working sample shall be quartered and ground in an agate mortar to a very nearly gritless powder. In the case of hard burned brick or hard lump chrome ore the very fine grinding assists materially in solution of the sample.

Method. 21. Dry the fine, ground powder at 110° C. for not less than one hour. Cool in desiccator. Fuse 0.5 g. of the thoroughly dried sample in approximately 10 g. of fused potassium bi-sulfate in a platinum crucible. The usual cautions of a bi-sulfate fusion should be observed: that is, the flame turned very low so that the bi-sulfate just melts, increasing the flame so that the bi-sulfate remains in a liquid condition, very gradually increasing until the crucible reaches a dull red heat. The object of the slow heating is to retain the sulfuric acid, which is the effective part of the flux. The most refractory ores should be thoroughly fused in $1\frac{1}{2}$ to 2 hours. Remove the crucible from the burner and allow to cool in the air. The bi-sulfate melt will shrink from the crucible and may be removed easily in one cake. Dissolve the fusion in 200 cc. hot HCl (1 : 1), boiling until the solution acquires a clear, bright green color. Transfer the solution to a porcelain evaporating dish and evaporate to dryness on a water bath. Bake at exactly 120° C. until free from hydrochloric acid fumes. Moisten with hot HCl (1 : 1), evaporate on water bath and bake a second time at 120° C. Dissolve in 200 cc. hot dilute HCl (1 : 1), and filter out the silica. Ignite and weigh the silica in a platinum crucible. Add two drops concentrated H_2SO_4 and sufficient HF to remove all silica. Evaporate, ignite and weigh. The difference gives the amount of silica present in the sample. Should there be any appreciable residue left in the crucible it should be fused with a small amount of fused $KHSO_4$ and added to the filtrate from the silica.

[1] For detailed specifications for these sieves, see the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

Dilute the filtrate from the silica to about 500 cc. and precipitate iron, aluminum, chromium and titanium, if present, with NH_4OH, using care to have not more than two drops of NH_4OH in excess in order to avoid solution of chromium and aluminum hydroxide in the alkaline solution. On account of the relatively large amount of magnesium that is usually present in chrome ores and chrome brick it is advisable to dissolve the precipitate in hot dilute HCl and precipitate a second time. Filter out the combined hydroxides and dissolve them on the filter paper with HNO_3, catching the solution in a porcelain casserole. Add a few crystals of $KClO_3$ and evaporate the nitric solution, adding a crystal of $KClO_3$ from time to time until the chromium is completely oxidized, which is indicated by a change from a dull green to a brilliant orange color. Dilute this solution with hot water and again precipitate with NH_4OH, filtering as before. The filtrate contains a large percentage of the chromium, while the residue on the filter consists of iron and aluminum hydroxides and titanium, if present. Repeat the oxidation two or three times until the filtrate from the iron and aluminum hydroxide precipitation shows no further traces of chromium. Concentrate and combine the filtrates containing chromium to approximately 500 cc. Add H_2SO_3 until the liquid changes from a bright orange to a brilliant green color and heat to boiling. Add NH_4OH to precipitate chromium as hydroxide, having not more than two drops in excess, as stated above. Filter, wash well with hot water, ignite and weigh as Cr_2O_3. The iron and aluminum and titanium hydroxides are ignited and weighed in a platinum crucible. Fuse the combined oxides with a small amount of $KHSO_4$, following the same precautions as mentioned above. Dissolve the fusion in dilute H_2SO_4, reduce with H_2S and titrate the iron with standard permanganate solution. Determine the titanium present by the colorimetric method, as described in Section 16. Subtract the combined amounts of Fe_2O_3 and TiO_2 from the total weight of the three oxides and thus determine Al_2O_3. If titanium is absent, reduce the iron with metallic zinc and titrate with K_2MnO_4, which is a little quicker method than the H_2S reduction.

To the combined filtrates from the precipitations of iron aluminum, chromium and titanium hydroxides add bromine, allow to stand for a few minutes and then add sufficient NH_4OH to precipitate the manganese hydroxide. Boil to concentrate the manganese hydroxide if any be present. Filter, ignite and

weigh as Mn_3O_4. In the filtrate from the manganese add NH_4OH and $(NH_4)_2C_2O_4$. Heat to boiling and when the calcium oxide has settled, filter with the usual precautions. The calcium oxide may be determined by ignition or by the volumetric method. (Refer to the determination of calcium oxide as described in Section 17.)

Evaporate the filtrate from the calcium oxalate to dryness with HNO_3 to remove the excess of ammonium salts. Dissolve the residue in hot water, filter, allow to cool and determine MgO as described in Section 18.

Determine alkalies by the J. Lawrence Smith Method, as described in Section 19.

RAPID ROUTINE METHOD FOR THE ANALYSIS OF CHROME BRICK AND CHROME ORE.

Rapid Routine Method.

22. The sample shall be secured after the manner of any of the approved methods of sampling. If from ores or chrome bearing refractories it shall not be less than 10 lb. in small nut-sized particles when sent to the laboratory. If from chrome brick, the sample shall consist of a small portion from the interior of from 15 or 25 bricks. This 10-lb. sample shall be crushed to pass a No. 20 sieve[1] and quartered, until a 4-oz. sample is secured, which shall be ground to pass a No. 100 sieve.[1] From this portion a smaller working sample shall be quartered and ground in an agate mortar to a very nearly gritless powder. In the case of hard burned brick or hard lump chrome ore the very fine grinding assists materially in solution of the sample. Dry the fine, ground powder at 110° C. for not less than 1 hour. Cool in desiccator.

CHROMIUM.

Chromium.

23. Fuse 0.5 g. of the sample in 8 to 10 g. of sodium peroxide in a nickel or porcelain crucible over a Bunsen burner, being careful not to heat too strongly. When fused, leach out with cold water, boil 15 minutes to decompose any sodium peroxide. Cool and add 100 cc. of H_2SO_4 (1:1). Cool to tap-water temperature and titrate with ferrous ammonium sulfate and potassium permanganate. Time 30 to 45 minutes.

[1] For detailed specifications for these sieves, see the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

SILICA AND IRON.

Silica and Iron.

24. Fuse 0.5 g. of the sample in a nickel crucible over a Bunsen burner. Leach out with cold water, make acid with HCl and evaporate to dryness. Take up with 200 cc. water and add 10 cc. HCl. Filter off silica and wash well with hot water and hot dilute HCl. For high silica a second evaporation is necessary. The filtrate or combined filtrate is made alkaline with sodium hydroxide and about 5 cc. of bromine added. The mass is allowed to digest for $\frac{1}{2}$ hour on a steam bath. This oxidizes the iron and chromium, precipitates the iron and dissolves the chromium and aluminum. After precipitating the iron twice it is filtered off on an asbestos plug and washed well with hot water. This precipitate of iron may be dissolved off the plug with hot HCl (1:1) and determined by the stannous chloride reduction, and permanganate method.

A blank should be run in conjunction with this method and deductions made when necessary to determine the purity of the chemicals used.

STANDARD DEFINITIONS FOR CLAY REFRACTORIES[1]

Serial Designation: C 27 – 20

These definitions are issued under the fixed designation C 27; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1919; ADOPTED, 1920.

1. The following definitions relating to the resistance to heat and the constancy of volume of clay refractories are recommended for the purpose of classification:

Method of Determining Softening Point.

2. The softening point referred to in the following definitions shall be determined in accordance with the Standard Method of Test for Softening Point of Fire-Clay Brick (Serial Designation: C 24) of the American Society for Testing Materials.[2]

Test for Linear Contraction or Expansion.

3. The test for linear contraction or expansion referred to in the following definitions shall be conducted in accordance with the Standard Method of Test for Porosity and Permanent Volume Changes in Refractory Materials (Serial Designation: C 20) of the American Society for Testing Materials.[3]

HIGH HEAT DUTY BRICK

(*A*) *Clay Fire Brick.*

(Silica content less than 70 per cent.)

Softening Point.

4. The softening point of clay fire brick for high heat duty shall not be lower than that of standard cone No. 31 (about 1685° C., or 3065° F.).

Linear Contraction or Expansion.

5. When duplicate samples of clay fire brick for high heat duty are heated uniformly in a suitable furnace to a temperature of 1400° C. (2552° F.), maintained at this temperature for 5 hours, and cooled, they shall not show a contraction of more than 1.5 per cent of the original length nor an expansion of more than 1 per cent.

Option.

6. When a brick of this type softens at a temperature not below the softening point of standard cone No. 29 (about 1650° C., or 3002° F.) it may be tested according to classification (*B*) for

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] See p. 151. [3] See p. 148.

siliceous clay fire brick without losing in standing if it passes the tests.

(*B*) *Siliceous Clay Fire Brick.*

(Silica content 70 per cent or over.)

Softening Point.

7. The softening point of siliceous clay fire brick for high heat duty shall not be lower than that of standard cone No. 28 (about 1635° C., or 2975° F.).

Load Test.

8. All siliceous clay fire brick for high heat duty shall be subjected to a load test in accordance with the requirements of the Standard Method of Test for Refractory Materials under Load at High Temperatures (Serial Designation: C 16) of the American Society for Testing Materials.[1] The pressure to be applied upon the brick (placed on end) shall be 25 lb. per sq. in. and the maximum furnace temperature 1350° C. (2462° F.). The brick shall not show a contraction of more than 4 per cent of the original length, nor an expansion of more than 1 per cent.

Linear Contraction or Expansion.

9. When duplicate samples of siliceous clay fire brick for high heat duty are heated uniformly in a suitable furnace to a temperature of 1400° C. (2552° F.), maintained at this temperature for 5 hours, and cooled, they shall not show a contraction of more than 1.5 per cent of the original length nor an expansion of more than 1 per cent.

INTERMEDIATE HEAT DUTY BRICK

Softening Point.

10. The softening point of brick for intermediate heat duty shall not be lower than that of standard cone No. 28 (about 1635° C., or 2975° F.).

Linear Contraction or Expansion.

11. When duplicate samples of brick for intermediate heat duty are heated uniformly in a suitable furnace to a temperature of 1350° C. (2462° F.), maintained at this temperature for 5 hours, and cooled, they shall not show a contraction of more than 1.5 per cent of the original length nor an expansion of more than 1 per cent.

MODERATE HEAT DUTY BRICK

Softening Point.

12. The softening point of brick for moderate heat duty shall not be lower than that of standard cone No. 26 (about 1600° C., or 2912° F.).

Linear Contraction or Expansion.

13. When duplicate samples of brick for moderate heat duty are heated uniformly in a suitable furnace to a temperature of 1290° C. (2354° F.), maintained at this temperature for 5

[1] See p. 143.

hours, and cooled, they shall not show a contraction of more than 1.5 per cent of the original length nor an expansion of more than 1 per cent.

LOW HEAT DUTY BRICK

14. The softening point of brick for low heat duty shall not be lower than that of standard cone No. 19 (about 1510° C., or 2750° F.). **Softening Point.**

STANDARD SPECIFICATIONS
FOR
CLAY SEWER PIPE[1]

Serial Designation: C 13 - 24

These specifications are issued under the fixed designation C 13; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1917; ADOPTED IN AMENDED FORM, 1920; REVISED, 1924.

Scope.

1. These specifications cover clay products intended to be used for the conveyance of sewage, industrial wastes and storm water.

Single Class.

2. Sewer pipes furnished under these specifications shall be of a single class to be designated "A.S.T.M. Clay Sewer Pipe."

MATERIAL AND MANUFACTURE

Materials.

3. (*a*) Clay pipes shall be manufactured from surface clay, fire clay or shale, or a combination of these materials.

(*b*) *Surface clay* is an unconsolidated, unstratified clay,[2] occurring on the surface.

(*c*) *Fire clay* is a sedimentary clay[2] of low flux content.

NOTE.—It is usually associated with coal measures.

(*d*) *Shale* is a thinly stratified, consolidated sedimentary clay[2] with well marked cleavage parallel to the bedding.

(*e*) The materials shall possess such physical and chemical properties that when molded into pipes and subjected to a suitable temperature the product will be strong, durable and serviceable, free from objectionable defects, and in compliance with these specifications and tests.

CHEMICAL TESTS AND REQUIREMENTS

Chemical Tests and Requirements.

4. The consumer or purchaser may prescribe in advance special chemical requirements in cases where industrial wastes have marked acid or alkaline character, or are of abnormally high temperatures. He may make use of chemical analysis of the pipe material to ascertain whether these special requirements are met. The presence of

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-4 on Clay and Cement-Concrete Pipe.

[2] The definitions for the terms "surface clay," "fire clay," and "shale" are based upon the following definition for the term "clay":

Clay.—An earthy or stony mineral aggregate consisting essentially of hydrous silicates of alumina, plastic when sufficiently pulverized and wetted, rigid when dry and vitreous when burned at a sufficiently high temperature.

visible grains or masses of caustic lime, iron pyrites or any other materials which cause slaking or disintegration shall be a cause for rejection.

PHYSICAL TESTS

5. The physical tests of pipes shall include: crushing test, hydrostatic pressure test, and absorption test. Physical Tests.

6. The specimens to be tested shall be selected by the purchaser or his representative at the point or points designated by him when placing the order. The manufacturer or seller shall furnish specimens for test, without separate charge, up to 1 per cent of the number of pipes to be delivered or furnished in each size of pipe, except that in no case shall the number of specimens furnished be less than five. Test Specimens.

7. (*a*) Failure of 20 per cent of the specimens to meet the requirements of any of the tests imposed, shall result in rejection of all the pipe in the shipment or delivery, corresponding to the sizes thus failing to comply; except that in the event of 20 per cent of the specimens in any size failing to meet the requirements, the manufacturer or seller may, with the consent of the consumer or purchaser, furnish for test, without charge, additional specimens from the same shipment to be selected as specified in Section 6. In case more than 80 per cent of the specimens tested, including those first tested, shall show substantial compliance for each of the various tests performed, then the entire shipment or delivery for this size shall be accepted; otherwise it shall be rejected. Acceptance or Rejection on Result of Tests.

(*b*) In addition to the foregoing requirements, failure of individual specimens to develop 75 per cent of the average crushing strength requirements shall be cause for rejection of the shipment, but the seller may cull the pipe and submit the balance of the shipment for retest, and, if the shipment then passes all of the requirements of these specifications, it shall be accepted.

8. The specimens of pipes shall be sound, full-size pipe. They shall first be freed from all visible moisture. When dry, each specimen shall be weighed, measured and inspected. The results of these observations shall be recorded and preserved as shown in Table I. Measurement and Observation of Specimens.

9. Specimens which, when placed in a vertical position, do not give a metallic ring when struck with a hammer, or are observed to have fire cracks or other defects in form or dimensions in excess of the limits permitted in these specifications, shall be discarded and replaced with additional specimens from the shipment. Rejection of Defective Specimens.

(A) Crushing Tests

10. (*a*) Any prime mover or hand power which will apply the load at a uniform rate of about 2000 lb. per minute, or in increments Application of Load.

TABLE I.—DIMENSIONS, ETC., OF CLAY SEWER PIPE FURNISHED AS TEST SPECIMENS.

Specimen No.	Date.	Job No.	Material.	Manufacturer.	Location of Factory.	Nominal Size, in.	Weight, lb.	Length, ft.	Thickness of Spigot, in.				Thickness of Socket, in.				Internal Diameter, in.						Remarks.
									Invert.	Crown.	Side.	Side.	Invert.	Crown.	Side.	Side.	Spigot.		Back of Socket.		Socket.		
																	Vertical.	Horizontal.	Vertical.	Horizontal.	Vertical.	Horizontal.	

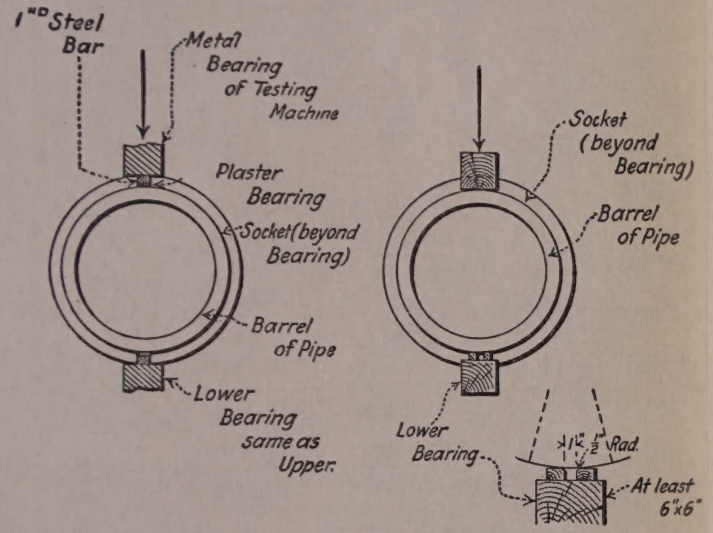


FIG. 1.—Knife or Two-Edge Bearings.

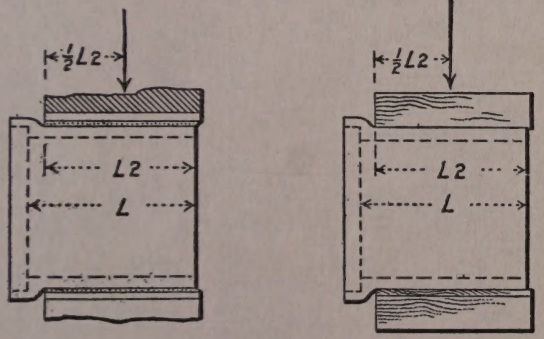


FIG. 2.—Three-Edge Bearings.

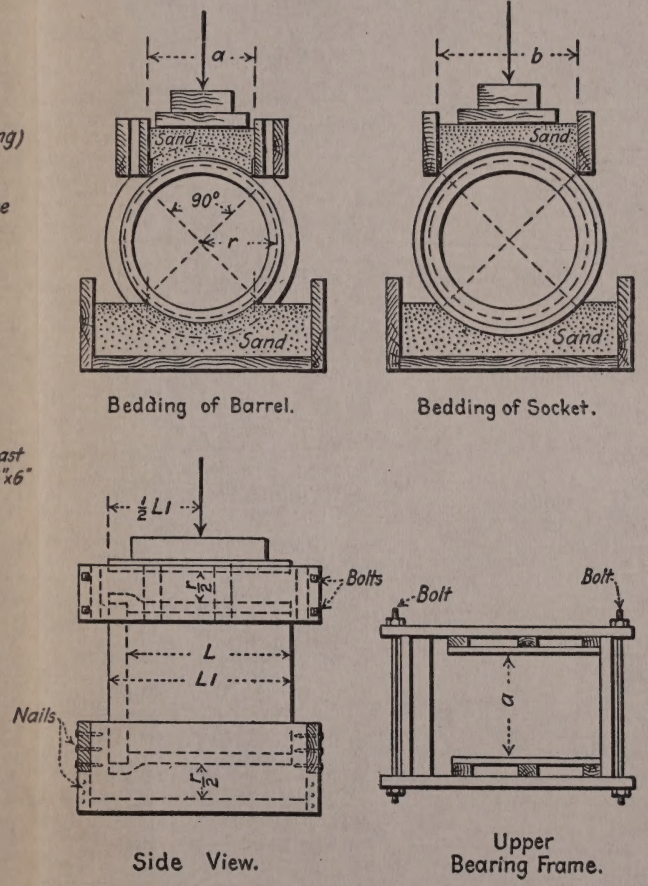
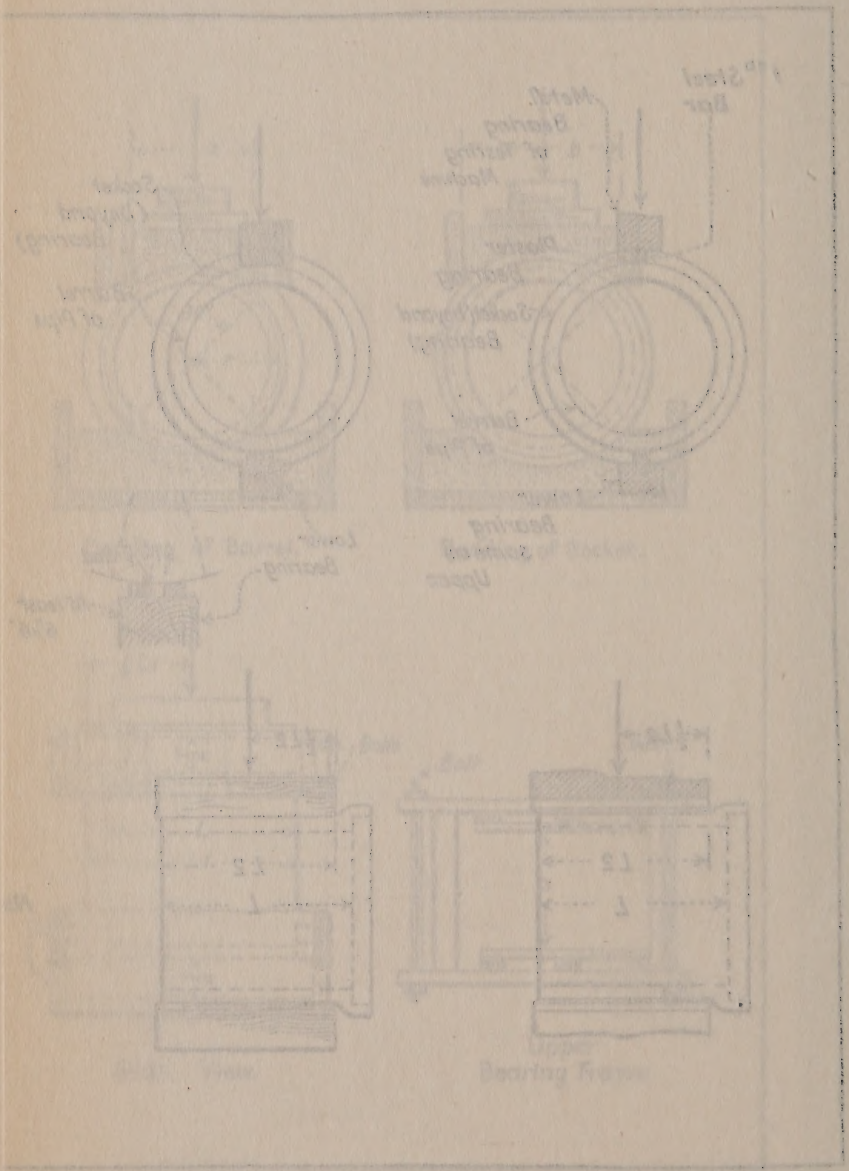


FIG. 3.—Sand Bearings.



of not more than 100 lb. at the same rate, may be used in making the test.

(*b*) The pipe shall not be allowed to stand under load longer than is required to apply the load and to observe and record it.

(*c*) The testing machine shall be substantial and rigid throughout, so that the distribution of the load will not be affected appreciably by the deformation or yielding of any part.

(*d*) The bearings and the specimen shall be accurately centered so as to secure a symmetrical distribution of the loading on each side of the center of the pipe in every direction.

(*e*) The load shall be applied until the pipe yields by cracks passing through the shell.

Knife or Two-Edge Bearings.

11. (See Fig. 1, Plate III.).—Except as otherwise hereinafter specified, the pipe to be tested shall be supported by a metallic knife bearing 1 in. wide and extended from a point just back of the socket to the spigot end of the pipe. Before the pipe is placed, a fillet of plaster of Paris and sand 1 in. wide, and thick enough to compensate for all the inequalities of the pipe barrel, shall be cast on the surface of the knife-edge bearing. The pipe shall be placed upon the fillet while the plaster of Paris is still somewhat plastic. The load shall be applied through an upper knife bearing of the same size and length as the lower bearing. A plaster-of-Paris fillet 1 in. wide shall be cast along the length of the crown of the pipe to equalize the lower bearing before the upper one is brought into contact.

Both of the bearings shall be sufficiently rigid to transmit and receive uniform loads throughout their lengths without deflection, and shall be so attached to the machine as to transmit and receive the maximum stresses produced by the tests without lost motion, vibration or sudden shock.

At the option of the consumer or purchaser the crushing test may be applied with sand bearings or with two or three-edge bearings.

The crushing strength shall be calculated by dividing the total load required to break each pipe by the net inside length of the barrel of the pipe, measuring from the bottom of the socket to the end of the spigot.

Three-Edge Bearings.

12. (See Fig. 2, Plate III.).—When three-edge bearings are used, the ends of each specimen of pipe shall be accurately marked in halves of the circumference prior to the test.

The two lower bearings shall consist of two wooden strips with vertical sides, each strip having its interior top corner rounded to a radius of approximately $\frac{1}{2}$ in. They shall be straight, and shall be

securely fastened to a rigid block with their interior vertical sides 1 in. apart.

The upper bearing shall be a wooden block, straight and true from end to end.

The test load shall be applied through the upper bearing block in such a way as to leave the bearing free to move in a vertical plane passing midway between the lower bearings.

In testing a pipe which is "out of straight," the lines of the bearings chosen shall be from those which appear to give most favorable conditions for fair bearings.

Sand Bearings.

13. (See Fig. 3, Plate III.).—When sand bearings are used the ends of each specimen of pipe shall be accurately marked prior to the test in quarters of the circumference. Specimens shall be carefully bedded, above and below, in sand, for one-fourth the circumference of the pipe measured on the middle line of the barrel. The depth of bedding above and below the pipe at the thinnest points shall be one-half the radius of the middle line of the barrel.

The sand used shall be clean, and shall be such as will pass a No. 4 sieve.

The top bearing frame shall not be allowed to come in contact with the pipe nor with the top bearing plate. The upper surface of the sand in the top bearing shall be struck level with a straight edge, and shall be covered with a rigid top bearing plate, with lower surface a true plane, made of heavy timbers or other rigid material, capable of distributing the test load uniformly without appreciable bending. The test load shall be applied at the exact center of this top bearing plate, in such a manner as to permit free motion of the plate in all directions. For this purpose a spherical bearing is preferred, but two rollers at right angles may be used. The test may be made without the use of a testing machine, by piling weights directly on a platform resting on the top bearing plate, provided, however, that the weights shall be piled symmetrically about a vertical line through the center of the pipe, and that the platform shall not be allowed to touch the top bearing frame.

The frames of the top and bottom bearings shall be made of timbers so heavy as to avoid appreciable bending by the side pressure of sand. The interior surfaces of the frames shall be dressed. No frame shall come in contact with the pipe during the test. A strip of cloth may, if desired, be attached to the inside of the upper frame on each side, along the lower edge, to prevent the escape of sand between the frame and the pipe.

Number of Specimens.

14. The crushing test shall ordinarily be applied to not less than 75 per cent of the specimens received for testing purposes.

15. Pieces of the crushed pipe may be used as specimens in making the absorption test. Specimens for Absorption Test.

(*B*) *Hydrostatic Test*

16. Sound full-size pipe not exceeding about 25 per cent of the specimens received for test in each size of pipe, shall be tested for leakage under internal hydrostatic pressure. Hydrostatic Test.

The ends of the pipe shall be tightly closed by wooden or metallic bulkheads or covers faced with rubber or leather so that no leakage shall occur through the covers at the test pressure. One cover shall be provided with a $\frac{3}{4}$-in. wrought-iron nipple passing through the cover, and held securely in place and made water-tight by means of locknuts and washers or gaskets. The outer end of the nipple shall be connected with a pump discharge or water service line.

Water pressure, as measured by a standardized gage attached to the delivery pipe close to the specimen, shall be internally applied to the specimen as follows:

5 lb. per sq. in. for 5 minutes.
10 " " " 10 "
15 " " " 15 "

The specimens shall show no leakage under these pressures. Moisture appearing on the surface of the pipe in the form of patches or beads, adhering to the surface, shall not be considered leakage.

(*C*) *Absorption Test*

17. The specimens shall be sound pieces, with all edges broken, and may be from pipes broken in the crushing or other tests. They shall be from 12 to 20 sq. in. in area, and shall be as nearly square as they can be readily prepared. They shall be free from observable cracks, fissures, laminations or shattered edges. Test Specimens.

18. Preparatory to the absorption test, the specimen shall be first weighed and then dried in a drier or oven at a temperature of not less than 110° C. (230° F.) for not less than three hours. After removal from the drier, the specimen shall be allowed to cool in dry air to a temperature of 20 to 25° C. (68 to 77° F.), and then reweighed. Drying.

If the specimen is comparatively dry when taken, and the second weight closely agrees with the first, it shall be considered dry. If the specimen is wet when taken it shall be placed in the drier for a drying treatment of two hours and reweighed. If the third weight checks the second the specimen shall be considered dry. In case of any doubt, the specimen shall be redried for two-hour periods, until check weights are obtained.

Weighing. 19. The balance used shall be sensitive to 0.5 g. when loaded with 1 kg., and weighings shall be read to the nearest gram. When other than metric weights are used, the same degree of accuracy shall be obtained.

Immersion. 20. The specimen, after final drying, cooling and weighing, shall be placed with other similar specimens in a suitable wire receptacle, packed tightly enough to prevent jostling, covered with distilled water or rainwater, raised to the boiling point and boiled for five hours, and then cooled in water to a final temperature of 10 to 15° C. (50 to 59° F.).

Reweighing. 21. The specimen shall be allowed to drain for one minute, and the superficial moisture having been removed by towel or blotting paper, placed upon the balance.

Calculation of Absorption. 22. The test result shall be calculated as percentage of the initial dry weight.

Number of Specimens. 23. One specimen shall be tested of each pipe broken in the crushing test.

Reporting Results. 24. The results shall be reported separately for each individual specimen, together with the mean for all the specimens from the same shipment of pipe.

Identification. 25. Each specimen shall be marked so that it may be identified with the pipe used in the crushing test from which the specimen was taken. The marking shall be applied so that the pigment used shall not cover more than 1 per cent of the total superficial area of the specimen.

PHYSICAL TEST REQUIREMENTS

Physical Requirements. 26. The test requirements of clay sewer pipe shall be as given in Table II. The individual results of the various tests for each size of pipe and for each shipment class and mill shall be tabulated separately so as to show the percentage which fails to comply with the requirements of each test.

A.S.T.M. SIZES AND DIMENSIONS

Sizes and Dimensions. 27. Pipes shall be furnished of the sizes, internal diameter, and with the dimensions given in Tables III and IV. Where several lengths are mentioned in the table the consumer or purchaser shall indicate, at the time of purchase, which lengths shall be furnished; and unless so indicated, the manufacturer shall furnish such lengths as he may elect.

Permissible Variations. 28. The permissible variation from the dimensions given in Table III shall not exceed those stated in Table IV. Where the thickness of barrel is increased beyond that given in Table IV in order to meet

the specified requirements of strength, the diameter at inside of socket shall be increased by double the increase in thickness of barrel. Pipes intended to be straight shall not have variation in alignment of more than $\frac{1}{8}$ in. per foot of length.

WORKMANSHIP AND FINISH

Absence of Defects.

29. Pipes shall be substantially free from fractures, large or deep cracks and blisters, laminations and surface roughness.

TABLE II.—PHYSICAL TEST REQUIREMENTS OF CLAY SEWER PIPE.

Internal Diameter, in.	Average Crushing Strength, lb. per lin. foot.		Maximum Absorption, per cent.
	Knife-Edge[1] and Three-Edge[2] Bearings.	Sand Bearings.[3]	
4	1000	1430	8
6	1000	1430	8
8	1000	1430	8
10	1100	1570	8
12	1200	1710	8
15	1370	1960	8
18	1540	2200	8
21	1810	2590	8
24	2150	3070	8
27	2360	3370	8
30	2580	3690	8
33	2750	3930	8
36	3080	4400	8
39	3300	4710	8
42	3520	5030	8

[1] See Section 11. [2] See Section 12. [3] See Section 13.

Scored Ends.

30. The inner surface of the socket and the outer surface of the spigot end shall be scored by triangular shaped or semi-circular shaped rings about $\frac{1}{8}$ in. deep.

The number of scorings shall be as follows:

For pipes	4	to	6	in.	in internal diameter, inclusive	.....	none
" "	8	"	10	"	" " "	.....	2
" "	12	"	21	"	" " "	.....	3
" "	24	"	30	"	" " "	.....	4
" "	33	"	42	"	" " "	.....	5

Salt Glaze.

31. The glaze shall consist of a continuous layer of bright or semi-bright glass substantially free from coarse blisters and pimples. If present, none of these shall project more than $\frac{1}{8}$ in. above the surrounding surface. Not more than 10 per cent of the inner surface of any pipe barrel shall be bare of glaze except the socket, where it may be entirely absent. Glazing will not be required on the outer surface of the barrel at the spigot end for a distance from the end equal to two-thirds the specified depth of socket for the corresponding size of pipe. Where glazing is required there shall be absence of any well-defined net work of crazing lines or hair cracks. All glazing shall be equal to that produced by the best salt-glazing process.

Finish of Ends. 32. The ends of the pipes shall be square with their longitudinal axis, except as provided in Table IV.

Specials. 33. (*a*) Special shapes shall have a plain spigot end and a socket end corresponding in all respects with the dimensions specified for pipes of the corresponding internal diameter. Branch pipes shall be furnished to lay the same lengths as straight pipe. All specials shall conform in finish to the specifications for pipes given in Sections 29, 30, 31 and 32.

(*b*) Slants shall have their spigot ends cut at an angle of approximately 45 deg. with the longitudinal axis.

(*c*) Curves shall be at angles of 90, 45, $22\frac{1}{2}$ deg., as required. They shall conform substantially to the curvature specified.

TABLE III.—DIMENSIONS OF CLAY SEWER PIPE.

Internal Diameter (*D*), in.	Laying Length (*L*), ft.	Inside Diameter at Mouth of Socket (D_s), in.[1]	Depth of Socket (L_s), in.	Minimum Taper of Socket (*H*)	Thickness of Barrel (*T*), in.	Thickness of Socket (T_s)
4	2	6	$1\frac{1}{2}$	1:20	$\frac{9}{16}$	The thickness of the socket $\frac{1}{4}$ in. from its outer end shall not be less than three-fourths of the thickness of the barrel of the pipe
6	2	$8\frac{1}{4}$	2	1:20	$\frac{5}{8}$	
8	2, $2\frac{1}{2}$, 3	$10\frac{3}{4}$	$2\frac{1}{4}$	1:20	$\frac{3}{4}$	
10	2, $2\frac{1}{2}$, 3	13	$2\frac{1}{2}$	1:20	$\frac{7}{8}$	
12	2, $2\frac{1}{2}$, 3	$15\frac{1}{4}$	$2\frac{1}{2}$	1:20	1	
15	2, $2\frac{1}{2}$, 3	$18\frac{3}{4}$	$2\frac{1}{2}$	1:20	$1\frac{1}{4}$	
18	2, $2\frac{1}{2}$, 3	$22\frac{1}{4}$	3	1:20	$1\frac{1}{2}$	
21	2, $2\frac{1}{2}$, 3	26	3	1:20	$1\frac{3}{4}$	
24	2, $2\frac{1}{2}$, 3	$29\frac{1}{2}$	3	1:20	2	
27	$2\frac{1}{2}$, 3	$33\frac{1}{4}$	$3\frac{1}{2}$	1:20	$2\frac{1}{4}$	
30	$2\frac{1}{2}$, 3	37	$3\frac{1}{2}$	1:20	$2\frac{3}{8}$	
33	$2\frac{1}{2}$, 3	$40\frac{1}{4}$	4	1:20	$2\frac{5}{8}$	
36	$2\frac{1}{2}$, 3	44	4	1:20	$2\frac{3}{4}$	
39	$2\frac{1}{2}$, 3	$47\frac{1}{4}$	4	1:20	$2\frac{7}{8}$	
42	$2\frac{1}{2}$, 3	51	4	1:20	3	

[1] When pipes are furnished having an increase in thickness over that given in last column, the diameter of socket shall be increased by an amount equal to twice the increase of thickness of barrel.

(*d*) Branches shall be furnished with the connection or connections of the size or sizes specified, securely and completely fastened by fusion in the process of vitrification to the barrel of the pipe. T-branches and double T-branches shall have their axes perpendicular to the longitudinal axis of the pipe. Y-branches, double Y-branches, and V-branches shall have their axes approximately 45 deg. from the longitudinal axis of the pipe measured from the socket end. All branches shall terminate in sockets and the barrel of the branch shall be of sufficient length to permit making a proper joint when the connecting pipe is inserted in the branch socket.

(*e*) Channel or split pipes, curves and branches shall be accurate half sections of the corresponding size of straight pipe and specials.

MARKINGS

34. Pipes shall bear the initials or name of the person, company or corporation by whom they are manufactured, and the location of the mill. The markings shall be indented on the exterior of the barrel near the socket and shall be plainly legible for purpose of identification. Markings.

INSPECTION

35. All pipes shall be subject to inspection at the factory, trench or other point of delivery by a competent inspector employed by the consumer or purchaser. The purposes of the inspection shall be to cull and reject pipes which, independent of the physical tests herein specified, fail to comply with the requirements of these specifications. Inspection.

TABLE IV.—PERMISSIBLE VARIATIONS IN DIMENSIONS OF CLAY SEWER PIPE.

Normal Size, in.	Limits of Permissible Variation in:					
	Length, in. per ft. (−)	Lengths of Two Opposite Sides, in.	Internal Diameter, in.		Depth of Socket, in. (−)	Thickness of Barrel, in. (−)
			Spigot (±)	Socket (±)		
4	1/4	1/8	1/8	3/16	1/8	1/16
6	1/4	1/8	2/16	1/4	1/4	1/16
8	1/4	1/8	1/4	5/16	1/4	1/16
10	1/4	1/8	1/4	5/16	1/4	1/16
12	1/4	1/8	5/16	3/8	1/4	1/16
15	1/4	1/8	6/16	3/8	1/4	3/32
18	1/4	3/16	3/8	7/16	1/4	3/32
21	1/4	3/16	7/16	1/2	1/4	1/8
24	3/8	1/4	1/2	9/16	1/4	1/8
27	3/8	1/4	5/8	11/16	1/4	1/8
30	3/8	1/4	5/8	11/16	1/4	1/8
33	3/8	3/8	3/4	13/16	1/4	3/16
36	3/8	3/8	3/4	13/16	1/4	3/16
39	3/8	3/8	3/4	13/16	1/4	3/16
42	3/8	3/8	3/4	13/16	1/4	3/16

NOTE.—The minus sign (−) alone indicates that the plus variation is not limited; the plus and minus sign (±) indicates variation in both excess and deficiency in dimension.

36. Pipes shall be subject to rejection on account of the following: Rejection.

(*a*) Variations in any dimension exceeding the permissible variations given in Table IV.

(*b*) Fracture or cracks passing through the shell or socket, except that a single crack at either end of a pipe not exceeding 2 in. in length or a single fracture in the socket not exceeding 3 in. in width nor 2 in. in length will not be deemed cause for rejection unless these defects exist in more than 5 per cent of the entire shipment or delivery.

(*c*) Blisters where the glazing is broken or which exceed 3 in. in diameter, or which project more than 1/8 in. above the surface.

(*d*) Laminations which indicate extended voids in the pipe material.

(*e*) Fire cracks or hair cracks sufficient to impair the strength, durability or serviceability of the pipe.

(*f*) Variation of more than $\frac{1}{8}$ in. per linear foot in alignment of a pipe intended to be straight.

(*g*) Glaze which does not fully cover and protect all parts of the shell and ends except those exempted in Section 31; also glaze which is not equal to best salt glaze.

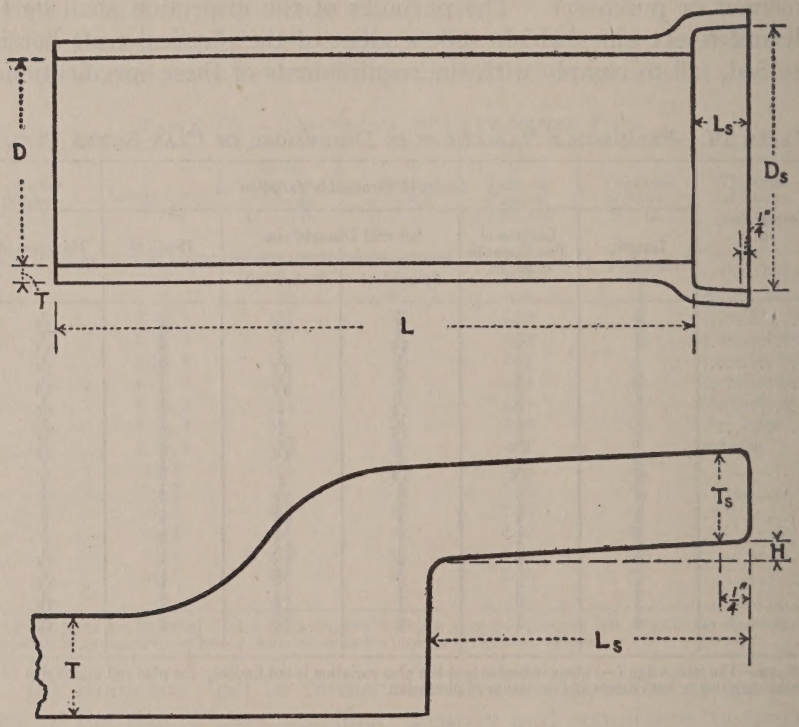


FIG. 4.—Application of Dimensions given in Table III.

(*h*) Failure to give a clear ringing sound when placed on end and dry-tapped with a light hammer.

(*i*) Insecure attachment of branches on spurs.

Marking of Rejected Specimens.

37. All rejected pipes shall be plainly marked by the inspector and shall be replaced by the manufacturer or seller with pipes which meet the requirements of these specifications, without additional cost to the consumer or purchaser.

STANDARD SPECIFICATIONS
FOR
CEMENT-CONCRETE SEWER PIPE[1]

Serial Designation: C 14 – 24

These specifications are issued under the fixed designation C 14; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1917; ADOPTED IN AMENDED FORM, 1920; REVISED, 1924.

Scope.

1. These specifications cover cement-concrete products intended to be used for the conveyance of sewage, industrial wastes and storm water.

Single Class.

2. Pipes furnished under these specifications shall be of a single class to be designated "A.S.T.M. Cement-Concrete Sewer Pipe."

MATERIAL AND MANUFACTURE

Materials.

3. (*a*) Cement-concrete pipes shall be manufactured from portland-cement concrete.

(*b*) *Concrete.*—By concrete is meant a suitable mixture of portland cement, mineral aggregates and water, hardened by hydraulic chemical action.

(*c*) *Cement.*—Portland cement shall meet the requirements of the Standard Specifications and Tests for Portland Cement (Serial Designation: C 9) of the American Society for Testing Materials.[2]

(*d*) The materials shall possess such physical and chemical properties that when molded into pipes and properly cured the product will be strong, durable and serviceable, free from objectionable defects, and in compliance with these specifications and tests.

CHEMICAL TESTS AND REQUIREMENTS

Chemical Tests and Requirements.

4. The consumer or purchaser may prescribe in advance special chemical requirements in cases where sewage, industrial wastes or ground waters have marked acid or alkaline character, or are of abnormally high temperatures. He may make use of chemical analysis of the pipe materials to ascertain whether these special

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-4 on Clay and Cement-Concrete Pipe.

[2] See p. 23.

requirements are met. The presence of deleterious materials causing slaking or disintegration shall be cause for rejection.

PHYSICAL TESTS

Physical Tests.

5. The physical tests of pipes shall include: crushing test, hydrostatic pressure test and absorption test.

Test Specimens.

6. The specimens to be tested shall be selected by the purchaser or his representative at the point or points designated by him when placing the order. The manufacturer or seller shall furnish specimens for test, without separate charge, up to 1 per cent of the number of pipes to be delivered or furnished in each size of pipe, except that in no case shall the number of specimens furnished be less than five.

Acceptance or Rejection on Result of Tests.

7. (*a*) Failure of 20 per cent of the specimens to meet the requirements of any of the tests imposed, shall result in rejection of all the pipe in the shipment or delivery, corresponding to the sizes thus failing to comply; except that in the event of 20 per cent of the specimens in any size failing to meet the requirements, the manufacturer or seller may, with the consent of the consumer or purchaser, furnish for test, without charge, additional specimens from the same shipment to be selected as specified in Section 6. In case more than 80 per cent of the specimens tested, including those first tested, shall show substantial compliance for each of the various tests performed, then the entire shipment or delivery for this size shall be accepted; otherwise it shall be rejected.

(*b*) In addition to the foregoing requirements, failure of individual specimens to develop 75 per cent of the average crushing strength requirements shall be cause for rejection of the shipment, but the seller may cull the pipe and submit the balance of the shipment for retest, and, if the shipment then passes all of the requirements of these specifications, it shall be accepted.

Measurement and Observation of Specimens.

8. The specimens of pipes shall be sound, full-size pipe. They shall first be freed from all visible moisture. When dry, each specimen shall be weighed, measured and inspected. The results of these observations shall be recorded and preserved as shown in Table I.

Rejection of Defective Specimens.

9. Specimens which, when placed in a vertical position, do not give a metallic ring when struck with a hammer, or are observed to have cracks or other defects in form or dimensions in excess of the limits permitted in these specifications, shall be discarded and replaced with additional specimens from the shipment.

(*A*) *Crushing Test*

Application of Load.

10. (*a*) Any prime mover or hand power which will apply the load at a uniform rate of about 2000 lb. per minute, or in increments

TABLE I.—DIMENSIONS, ETC., OF CEMENT-CONCRETE SEWER PIPE FURNISHED AS TEST SPECIMENS.

Specimen No.	Date.	Job No.	Material.	Manufacturer.	Location of Factory.	Nominal Size, in.	Weight, lb.	Length, ft.	Thickness of Spigot, in.				Thickness of Socket, in.				Internal Diameter, in.						Remarks.
																	Spigot		Back of Socket.		Socket.		
									Invert.	Crown.	Side.	Side.	Invert.	Crown.	Side.	Side.	Vertical.	Horizontal.	Vertical.	Horizontal.	Vertical.	Horizontal.	

of not more than 100 lb. at the same rate, may be used in making the test.

(*b*) The pipe shall not be allowed to stand under load longer than is required to apply the load and to observe and record it.

(*c*) The testing machine shall be substantial and rigid throughout, so that the distribution of the load will not be affected appreciably by the deformation or yielding of any part.

(*d*) The bearings and the specimen shall be accurately centered so as to secure a symmetrical distribution of the loading on each side of the center of the pipe in every direction.

(*e*) The load shall be applied until the pipe yields by cracks passing through the shell.

Knife or Two-Edge Bearings.

11. (See Fig. 1, Plate III.)[1]—Except as otherwise hereinafter specified, the pipe to be tested shall be supported by a metallic knife bearing 1 in. wide and extended from a point just back of the socket to the spigot end of the pipe. Before the pipe is placed a fillet of plaster of Paris and sand 1 in. wide, and thick enough to compensate for all the inequalities of the pipe barrel, shall be cast on the surface of the knife bearing. The pipe shall be placed upon the fillet while the plaster of Paris is still somewhat plastic. The load shall be applied through an upper knife bearing of the same size and length as the lower bearing. A plaster-of-Paris fillet 1 in. wide shall be cast along the length of the crown of the pipe to equalize the lower bearing before the upper one is brought into contact.

Both of the bearings shall be sufficiently rigid to transmit and receive uniform loads throughout their lengths without deflection, and shall be so attached to the machine as to transmit and receive the maximum stresses produced by the tests without lost motion, vibration or sudden shock.

At the option of the consumer or purchaser the crushing test may be applied with sand bearings or with two or three-edge bearings.

The crushing strength shall be calculated by dividing the total load required to break each pipe by the net inside length of the barrel of the pipe, measuring from the bottom of the socket to the end of the spigot.

Three-Edge Bearings.

12. (See Fig. 2, Plate III.)[1]—When three-edge bearings are used, the ends of each specimen of pipe shall be accurately marked in halves of the circumference prior to the test.

The two lower bearings shall consist of two wooden strips with vertical sides, each strip having its interior top corner rounded to a radius of approximately $\frac{1}{2}$ in. They shall be straight, and shall be

[1] See p. 170.

securely fastened to a rigid block with their interior vertical sides 1 in. apart.

The upper bearing shall be a wooden block, straight and true from end to end.

The test load shall be applied through the upper bearing block in such a way as to leave the bearing free to move in a vertical plane passing midway between the lower bearings.

In testing a pipe which is "out of straight," the lines of the bearings chosen shall be from those which appear to give most favorable conditions for fair bearings.

Sand Bearings.

13. (See Fig. 3, Plate III).[1]—When sand bearings are used, the ends of each specimen of pipe shall be accurately marked prior to the test in quarters of the circumference. Specimens shall be carefully bedded, above and below, in sand, for one-fourth the circumference of the pipe measured on the middle line of the barrel. The depth of bedding above and below the pipe at the thinnest points shall be one-half the radius of the middle line of the barrel.

The sand used shall be clean, and shall be such as will pass a No. 4 sieve.

The top bearing frame shall not be allowed to come in contact with the pipe nor with the top bearing plate. The upper surface of the sand in the top bearing shall be struck level with a straight edge, and shall be covered with a rigid top bearing plate, with lower surface a true plane, made of heavy timbers or other rigid material, capable of distributing the test load uniformly without appreciable bending. The test load shall be applied at the exact center of this top bearing plate, in such a manner as to permit free motion of the plate in all directions. For this purpose a spherical bearing is preferred, but two rollers at right angles may be used. The test may be made without the use of a testing machine, by piling weights directly on a platform resting on the top bearing plate, provided, however, that the weights shall be piled symmetrically about a vertical line through the center of the pipe, and that the platform shall not be allowed to touch the top bearing frame.

The frames of the top and bottom bearings shall be made of timbers so heavy as to avoid appreciable bending by the side pressure of sand. The interior surfaces of the frames shall be dressed. No frame shall come in contact with the pipe during the test. A strip of cloth may, if desired, be attached to the inside of the upper frame on each side, along the lower edge, to prevent the escape of sand between the frame and the pipe.

[1] See p. 170.

Number of Specimens.

14. The crushing test shall ordinarily be applied to not less than 75 per cent of the specimens received for testing purposes.

Specimens for Absorption Test.

15. Pieces of the crushed pipe may be used as specimens in making the absorption test.

(*B*) *Hydrostatic Test*

Hydrostatic Test.

16. Sound full-size pipe not exceeding about 25 per cent of the specimens received for test in each size of pipe, shall be tested for leakage under internal hydrostatic pressure.

The ends of the pipe shall be tightly closed by wooden or metallic bulkheads or covers faced with rubber or leather so that no leakage shall occur through the covers at the test pressure. One cover shall be provided with a $\frac{3}{4}$-in. wrought-iron nipple passing through the cover, and held securely in place and made water-tight by means of locknuts and washers or gaskets. The outer end of the nipple shall be connected with a pump discharge or water service line.

Water pressures, as measured by a standardized gage attached to the delivery pipe close to the specimen, shall be internally applied to the specimen as follows:

5	lb. per sq. in.	for	5	minutes
10	"	"	for 10	"
15	"	"	for 15	"

The specimens shall show no leakage under these pressures. Moisture appearing on the surface of the pipe in the form of patches or beads, adhering to the surface, shall not be considered leakage.

(*C*) *Absorption Test*

Test Specimens.

17. The specimens shall be sound pieces with all edges broken, and may be from pipes broken in the crushing or other tests. They shall be from 12 to 20 sq. in. in area, and shall be as nearly square as they can be readily prepared. They shall be free from observable cracks, fissures, laminations or shattered edges.

Drying.

18. Preparatory to the absorption test, the specimen shall be first weighed and then dried in a drier or oven at a temperature of not less than 110° C. (230° F.) for not less than three hours. After removal from the drier, the specimen shall be allowed to cool in dry air to a temperature of 20 to 25° C. (68 to 77° F.), and then reweighed.

If the specimen is comparatively dry when taken, and the second weight closely agrees with the first, it shall be considered dry. If the specimen is wet when taken it shall be placed in the drier for a drying treament of two hours and reweighed. If the third weight checks

the second the specimen shall be considered dry. In case of any doubt, the specimen shall be redried for two-hour periods, until check weights are obtained.

Weighing. 19. The balance used shall be sensitive to 0.5 g. when loaded with 1 kg., and weighings shall be read to the nearest gram. When other than metric weights are used, the same degree of accuracy shall be obtained.

Immersion. 20. The specimen, after final drying, cooling and weighing shall be placed with other similar specimens in a suitable wire receptacle, packed tightly enough to prevent jostling, covered with distilled water or rainwater, raised to the boiling point and boiled for five hours, and then cooled in water to a final temperature of 10 to 15° C. (50 to 59° F.).

TABLE II.—PHYSICAL TEST REQUIREMENTS OF CEMENT-CONCRETE SEWER PIPE

Internal Diameter, in.	Average Crushing Strength, lb. per lin. foot.		Maximum, Absorption per cent
	Knife-Edge[1] and Three-Edge[2] Bearings.	Sand Bearings.[3]	
4	1000	1430	8
6	1000	1430	8
8	1000	1430	8
10	1100	1570	8
12	1200	1710	8
15	1370	1960	8
18	1540	2200	8
21	1810	2590	8
24	2150	3070	8
27	2360	3370	8
30	2580	3690	8
33	2750	3930	8
36	3080	4400	8
39	3300	4710	8
42	3520	5030	8

[1] See Section 11. [2] See Section 12. [3] See Section 13.

Reweighing. 21. The specimen shall be allowed to drain for one minute, and, the superficial moisture having been removed by towel or blotting paper, placed upon the balance.

Calculation of Absorption. 22. The test result shall be calculated as percentage of the initial dry weight.

Number of Specimens. 23. One specimen shall be tested of each pipe broken in the crushing test.

Reporting Results. 24. The results shall be reported separately for each individual specimen together with the mean for all the specimens from the same shipment of pipe.

Identification. 25. Each specimen shall be marked so that it may be identified with the pipe used in the crushing test from which the specimen was taken. The marking shall be applied so that the pigment used shall

not cover more than 1 per cent of the total superficial area of the specimen.

PHYSICAL TEST REQUIREMENTS

Physical Test Requirements.

26. The test requirements of cement-concrete pipe shall be as given in Table II. The individual results of the various tests for each size of pipe and for each shipment class and mill shall be tabulated separately so as to show the percentage which fails to comply with the requirements of each test.

TABLE III.—DIMENSIONS OF CEMENT-CONCRETE SEWER PIPE.

Internal Diameter (D), in.	Laying Length (L), ft.	Inside Diameter at Mouth of Socket (D_s), in.[1]	Depth of Socket (L_s), in.	Minimum Taper of Socket (H)	Thickness of Barrel (T), in.	Thickness of Socket (T_s)
4	2, 2½	6	1½	1:20	9/16	The thickness of the socket ¼ in. from its outer end shall be not less than three-fourths of the thickness of the barrel of the pipe.
6	2, 2½	8¼	2	1:20	5/8	
8	2, 2½, 3	10¾	2¼	1:20	¾	
10	2, 2½, 3	13	2½	1:20	7/8	
12	2, 2½, 3	15¼	2½	1:20	1	
15	2, 2½, 3	18¾	2½	1:20	1¼	
18	2, 2½, 3	22¼	2¾	1:20	1½	
21	2, 2½, 3	26	2¾	1:20	1¾	
24	2, 2½, 3	29½	3	1:20	2⅛	
27	2½, 3	33¼	3¼	1:20	2¼	
30	2½, 3	37	3½	1:20	2½	
33	2½, 3	40¼	4	1:20	2¾	
36	2½, 3	44	4	1:20	3	
39	2½, 3	47¼	4	1:20	3¼	
42	2½, 3	51	4	1:20	3½	

[1] When pipes are furnished having an increase in thickness over that given in last column, then the diameter of socket shall be increased by an amount equal to twice the increase of thickness of barrel.

A.S.T.M. SIZES AND DIMENSIONS

Sizes and Dimensions.

27. Pipes shall be furnished of the sizes, internal diameter, and with the dimensions given in Tables III and IV. Where several lengths are mentioned in the table the consumer or purchaser shall indicate, at the time of purchase, which lengths shall be furnished; and unless so indicated, the manufacturer shall furnish such lengths as he may elect.

Permissible Variations.

28. The permissible variation from the dimensions given in Table III shall not exceed those stated in Table IV. Where the thickness of barrel is increased beyond that given in Table III in order to meet the specified requirements of strength, the diameter at inside of socket shall be increased by double the increase in thickness of barrel. Pipes intended to be straight shall not have variation in alignment of more than ⅛ in. per foot of length.

WORKMANSHIP AND FINISH

29. Pipes shall be substantially free from fractures, large or deep cracks and blisters, laminations and surface roughness. Absence of Defects.

30. The ends of the pipes shall be square with their longitudinal axis. Finish of Ends.

31. (*a*) Special shapes shall have a plain spigot end and a socket end corresponding in all respects with the dimensions specified for pipes of the corresponding internal diameter. Branches shall be furnished to lay the same lengths as straight pipe. All specials shall conform in finish to the specifications for pipes given in Sections 29 and 30. Specials.

TABLE IV.—PERMISSIBLE VARIATIONS IN DIMENSIONS OF CEMENT-CONCRETE SEWER PIPE.

Normal Size, in.	Limits of Permissible Variation in:				
	Length, in. per ft. (–)	Internal Diameter, in.		Depth of Socket, in. (–)	Thickness of Barrel, in. (–)
		Spigot (±)	Socket (±)		
4	$\frac{1}{4}$	$\frac{1}{8}$	$\frac{1}{8}$	$\frac{1}{8}$	$\frac{1}{16}$
6	$\frac{1}{4}$	$\frac{3}{16}$	$\frac{3}{16}$	$\frac{1}{4}$	$\frac{1}{16}$
8	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{1}{16}$
10	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{1}{16}$
12	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{1}{16}$
15	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{3}{32}$
18	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{3}{32}$
21	$\frac{1}{4}$	$\frac{5}{16}$	$\frac{5}{16}$	$\frac{1}{4}$	$\frac{1}{8}$
24	$\frac{3}{8}$	$\frac{5}{16}$	$\frac{5}{16}$	$\frac{1}{4}$	$\frac{1}{8}$
27	$\frac{3}{8}$	$\frac{5}{16}$	$\frac{5}{16}$	$\frac{1}{4}$	$\frac{1}{8}$
30	$\frac{3}{8}$	$\frac{3}{8}$	$\frac{3}{8}$	$\frac{1}{4}$	$\frac{1}{8}$
33	$\frac{3}{8}$	$\frac{3}{8}$	$\frac{3}{8}$	$\frac{1}{4}$	$\frac{3}{16}$
36	$\frac{3}{8}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{4}$	$\frac{3}{16}$
39	$\frac{3}{8}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{4}$	$\frac{3}{16}$
42	$\frac{3}{8}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{4}$	$\frac{3}{16}$

NOTE.—The minus sign (–) alone indicates that the plus variation is not limited; the plus and minus sign (±) indicates variation in both excess and deficiency in dimension.

(*b*) Slants shall have their spigot ends cut at an angle of approximately 45 deg. with the longitudinal axis.

(*c*) Curves shall be at angles of 90, 45, 22½ deg., as required. They shall conform substantially to the curvature specified.

(*d*) Branches shall be furnished with the connection or connections of the size or sizes specified, securely and completely fastened in the process of manufacture to the barrel of the pipe. T-branches and double T-branches shall have their axes perpendicular to the longitudinal axis of the pipe. Y-branches, double Y-branches, and V-branches shall have their axes approximately 45 deg. from the longitudinal axis of the pipe measured from the socket end. All branches

shall terminate in sockets and the barrel of the branch shall be of sufficient length to permit making a proper joint when the connecting pipe is inserted in the branch socket.

(*e*) Channel or split pipe, curves and branches shall be accurate half sections of the corresponding size of pipe or other specials.

MARKINGS

Markings.

32. Pipes shall bear the initials or name of the person, company or corporation by whom they are manufactured, and the location of

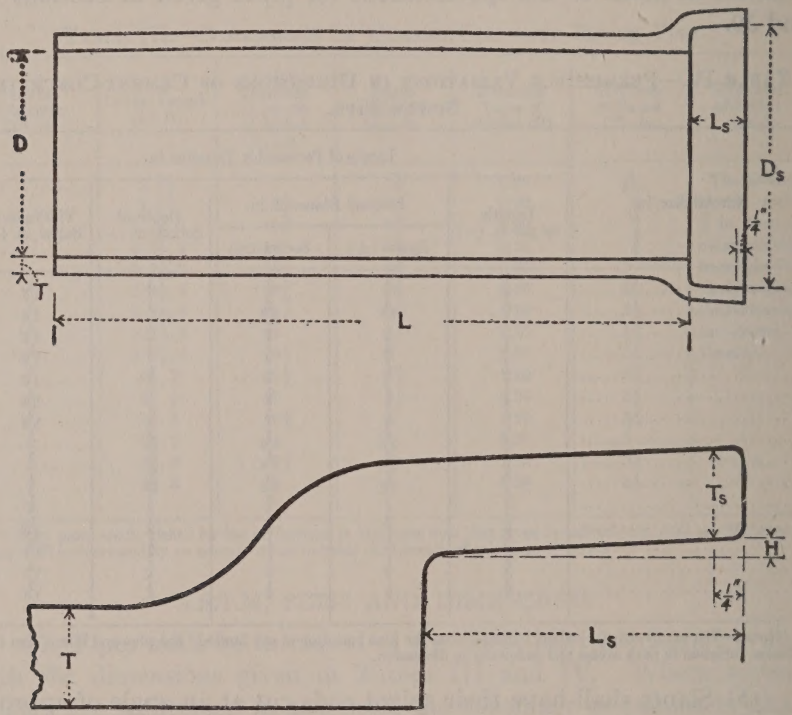


FIG. 4.—Application of Dimensions given in Table III.

the mill. The markings shall be indented or stenciled on the exterior or interior of the barrel near the socket and shall be plainly legible for purpose of identification.

INSPECTION

Inspection.

33. All pipes shall be subject to inspection at the factory, trench or other point of delivery by a competent inspector employed by the consumer or purchaser. The purposes of the inspection shall be to

cull and reject pipes which, independent of the physical tests herein specified, fail to meet the requirements of these specifications.

Rejection.

34. Pipes shall be subject to rejection on account of the following:

(*a*) Variations in any dimension exceeding the permissible variations given in Table IV.

(*b*) Fracture or cracks passing through the shell or socket, except that a single crack at either end of a pipe not exceeding 2 in. in length or a single fracture in the socket not exceeding 3 in. in width nor 2 in. in length will not be deemed cause for rejection unless these defects exist in more than 5 per cent of the entire shipment or delivery.

(*c*) Blisters where the surface is broken or which project more than $\frac{1}{8}$ in. above the surface.

(*d*) Defects which indicate imperfect mixing and molding.

(*e*) Cracks sufficient to impair the strength, durability or serviceability of the pipe.

(*f*) Variation of more than $\frac{1}{8}$ in. per linear foot in alignment of pipe intended to be straight.

(*g*) Failure to give a clear ringing sound when placed on end and dry-tapped with a light hammer.

(*h*) Insecure attachment of branches on spurs.

Marking of Rejected Specimens.

35. All rejected pipes shall be plainly marked by the inspector and shall be replaced by the manufacturer or seller with pipes which meet the requirements of these specifications, without additional cost to the consumer or purchaser.

STANDARD SPECIFICATIONS
FOR
DRAIN TILE[1]

Serial Designation: C 4 – 24.

These specifications are issued under the fixed designation C 4; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1914; REVISED, 1916, 1921, 1924.

These specifications were approved December 14, 1925, as "American Standard" by the American Engineering Standards Committee.

Scope.

1. (*a*) These specifications cover three classes of drain tile, namely, Farm Drain Tile, Standard Drain Tile, and Extra-Quality Drain Tile.

Classes.

(*b*) The purposes for which these classes are intended to be suitable are as follows:

Farm Drain Tile, for ordinary private drainage work on farms, for moderate sizes and depths;

Standard Drain Tile, for ordinary district land drainage at moderate depths;

Extra-Quality Drain Tile, for district land drainage, for considerable depths and where an extra quality is desired.

Basis of Purchase.

2. The purchaser shall specify the class or classes of tile to be supplied, whether Farm Drain Tile, Standard Drain Tile, or Extra-Quality Drain Tile. Standard Drain Tile shall be supplied where no other advance selection is stated.

Basis of Acceptance.

3. (*a*) The acceptability of drain tile shall be determined (1) by the results of the chemical and physical tests hereinafter specified, and (2) by visual inspection, to determine whether the tiles comply with the specifications as to dimensions, shape, and freedom from visible external and internal defects.

(*b*) The acceptance of drain tile as satisfactorily meeting one of these two general requirements shall not be construed as in any way waiving the other.

MATERIALS AND MANUFACTURE

Materials.

4. (*a*) These specifications shall apply to drain tile made of shale, fire clays or surface clays and to drain tile made of concrete.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-6 on Drain Tile.

(*b*) *Shale* is a thinly stratified, consolidated sedimentary clay[1] with well-marked cleavage parallel to the bedding.

(*c*) *Fire Clay* is a sedimentary clay[1] of low flux content.

NOTE.—It is usually associated with coal measures.

(*d*) *Surface Clay* is an unconsolidated, unstratified clay,[1] occurring on the surface.

(*e*) By concrete is meant a suitable mixture of portland cement, mineral aggregates and water, hardened by hydraulic chemical reaction.

(*f*) If the purchaser desires to exclude any of these materials he shall so specify in advance. All materials used shall be first-class of their kind and suitable for the purpose.

Manufacture.

5. The method of manufacture shall be such as to insure excellence of product and uniformity in quality.

CHEMICAL TESTS AND REQUIREMENTS

Chemical Tests and Requirements.

6. The purchaser may specify special chemical requirements as to resistance of the tile to chemical action in cases where soils or drainage waters have marked acid or alkaline character, or are of abnormally high temperature, and may prescribe chemical tests of the tile to ascertain whether these special requirements are met. Without a special agreement in advance, no drain tile shall be rejected by reason of its composition as determined by ultimate chemical analysis.

The presence in drain tile of visible grains or masses of caustic lime, iron pyrites, or any other minerals which are known to cause slaking or disintegration of the tile, shall be construed as a valid ground for rejection, unless satisfactory proof be submitted that the tiles are permanent and durable, and that the objectionable minerals are not present in quantity or condition to work damage.

PHYSICAL TESTS

Physical Tests.

7. The physical tests of drain tile shall include (*A*) Strength Tests and (*B*) Absorption Tests; and may include (*C*) Freezing and Thawing Tests, when specified by the purchaser in advance, or when called for by the manufacturer or other seller as provided in Sections 34, 35, 47 and 52.

Selection of Specimens of Tile.

8. The specimens of tile shall all be selected at the factory or at the shipping destination, or at the trench, at the

[1] The definitions for the terms "surface clay," "fire clay," and "shale" are based upon the following definition for the term "clay":

Clay.—An earthy or stony mineral aggregate consisting essentially of hydrous silicates of alumina, plastic when sufficiently pulverized and wetted, rigid when dry and vitreous when burned at a sufficiently high temperature.

option of the purchaser. The selection shall be made by a competent inspector employed by the purchaser. The inspector shall divide the tile into sub-classes if lack of uniformity in any important particular warrants such division, and shall select enough representative specimens of tile from each sub-class for a complete set of standard physical tests.

Number and Cost of Specimens of Tile.

9. A standard physical test shall comprise tests of five individual tiles. Specimens of tile may be selected by the inspector in such number as he judges necessary to determine fairly the quality of all the tile. The manufacturer or other seller shall furnish specimens of tile without separate charge up to 0.5 per cent of the whole number of tile, and the purchaser shall pay for all in excess of that percentage at the same rate as for other tile.

(*A*) *Strength Tests of Drain Tile.*

Tile Specimens.

10. The specimens of tile shall be unbroken, full-size tile.

Moisture Condition of Specimens of Tile.

11. The walls of the tile shall, at the time of testing, be as thoroughly wet as will result from completely covering with hay, cloth, or similar absorbent material, and keeping the covering wet for not less than 12 hours.

Temperature Condition of Specimens of Tile.

12. No specimen of tile shall be exposed to water or air temperatures lower than 40° F. from the beginning of wetting until tested. Frozen tile shall be completely thawed before the wetting begins.

Weighing.

13. Each specimen of tile shall be weighed on reliable scales just prior to testing, and the weights shall be reported.

Application of Load.

14. Any machine or hand method which will apply the load continuously, or in increments not exceeding 5 per cent of the estimated total breaking load, may be used in making the test. The tile shall not be allowed to stand under load longer than is required for observing and recording the loads. All solid parts of the bearing frames and bearing blocks shall be so rigid that the distribution of the load will not be affected appreciably by the deformation of any part. All bearings and the specimens of tile shall be so accurately centered as to secure a symmetrical distribution of the loading on each side of the center of the tile in every direction.

Choice of Bearings.

15. The purchaser shall choose (1) sand bearings, (2) two-edge bearings, or (3) three-edge bearings, for use in making strength tests of drain tile. (See Sections 18, 19, and 20.)

16. The test results shall be calculated and reported, in pounds per linear foot of tile, in terms of the "Ordinary Supporting Strength."[1] **Calculation and Reporting of Test Results.**

The ordinary supporting strength shall be calculated by multiplying the test breaking loads by the following factors: For sand bearings, 1.00; for two-edge bearings, 1.50; for three-edge bearings, 1.50.

The results of the strength tests shall be reported separately for each of the five individual specimens of tile constituting a standard test, together with the average.

17. The modulus of rupture may or may not be calculated and reported, at the option of the purchaser. When reported it shall be calculated by the equations:[2] **Modulus of Rupture.**

$$M = 0.20\, r \frac{W}{12} \quad \ldots\ldots\ldots\ldots\ldots\ldots \quad (1)$$

$$f = \frac{6M}{t^2} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (2)$$

where M = maximum bending moment in wall in pound-inches per inch of length, r = radius of middle line of tile wall in inches, W = ordinary supporting strength, calculated as prescribed in Section 16, in pounds per linear foot of tile, f = modulus of rupture in pounds per square inch, and t = thickness of tile wall in inches.

Five-eighths of the weight of the tile per linear foot for sand bearings, or three-fourths for two-edge or three-edge bearings, shall be added to W in computing the maximum bending moment M, when such addition exceeds 5 per cent of W. The value of t used shall be the average thickness of the wall at the top of the tile or that at the bottom, selecting the lesser of the two.

18. (See Fig. 1.)—When sand bearings are used, the ends of each specimen of tile shall be accurately marked in quarters of the circumference prior to the test. Specimens shall be carefully bedded, above and below, in sand, for one-fourth the circumference of the tile measured on the middle line of the wall. The depth of bedding above and below the tile at the thinnest points shall be one-half the radius of the middle line of the wall. **Sand Bearings.**

[1] The "ordinary supporting strength," when calculated as specified in Section 16, is approximately equal to the actual supporting strength of a tile when laid in a ditch by the "ordinary" method. See note under Table II.

[2] The coefficient of 0.20 in equation (1) approximates the value found by theoretical analysis and also that determined by extended tests.

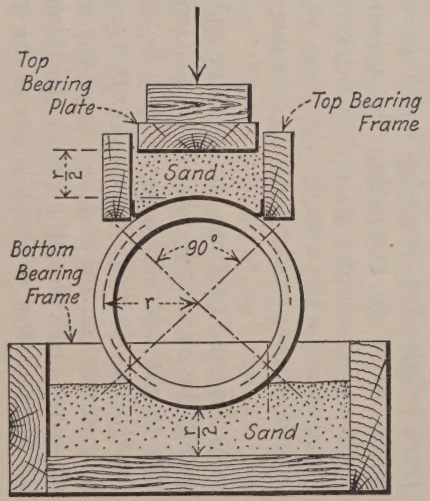


FIG. 1.—Sand Bearings.

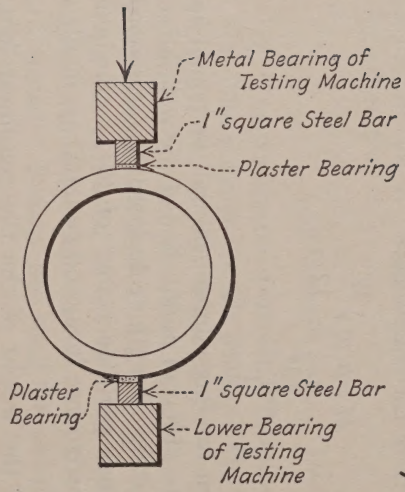
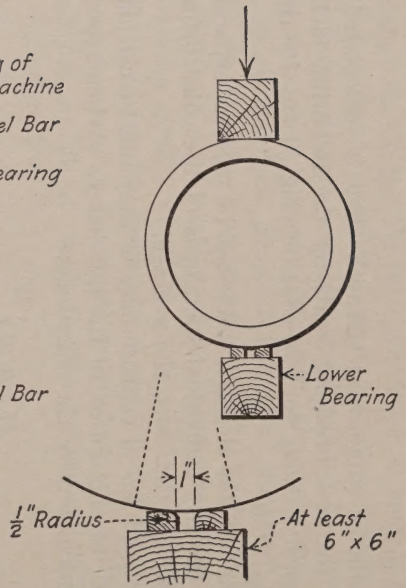


FIG. 2.—Two-Edge Bearings.



Detail of Lower Bearing.

FIG. 3.—Three-Edge Bearings.

The sand used shall be clean, and shall be such as will pass a No. 4 sieve.

The top bearing frame shall not be allowed to come in contact with the tile nor with the top bearing plate. The upper surface of the sand in the top bearing shall be struck level with a straight edge, and shall be covered with a rigid top bearing plate, with lower surface a true plane, made of heavy timbers or other rigid material, capable of distributing the test load uniformly without appreciable bending. The test load shall be applied at the exact center of this top bearing plate, in such a manner as to permit free motion of the plate in all directions. For this purpose a spherical bearing is preferred, but two rollers at right angles may be used. The test may be made without the use of a testing machine, by piling weights directly on a platform resting on the top bearing plate, provided, however, that the weight shall be piled symmetrically about a vertical line through the center of the tile, and that the platform shall not be allowed to touch the top bearing frame.

The frames of the top and bottom bearings shall be made of timbers so heavy as to avoid appreciable bending by the side pressure of the sand. The interior surfaces of the frames shall be dressed. No frame shall come in contact with the tile during the test. A strip of cloth may, if desired, be attached to the inside of the upper frame on each side, along the lower edge, to prevent the escape of sand between the frame and the tile.

Two-Edge Bearings.

19. (See Fig. 2.)—When two-edge bearings are used, the ends of each specimen of tile shall be accurately marked in halves of the circumference prior to the test.

The tile to be tested shall be supported by a metallic knife bearing 1 in. wide and extending the length of the tile. Before the tile is placed, a fillet of plaster-of-Paris and sand 1 in. wide, and thick enough to compensate for all the inequalities of the tile barrel, shall be cast on the surface of the knife-edge bearing. The tile shall be placed upon the fillet while the plaster-of-Paris is still somewhat plastic. The load shall be applied through an upper knife bearing of the same size and length as the lower bearing. A plaster-of-Paris fillet 1 in. wide shall be cast along the length of the crown of the tile to equalize the lower bearing before the upper one is brought into contact.

Both of the bearings shall be sufficiently rigid to transmit and receive uniform loads throughout their lengths without deflection, and shall be so attached to the machine as to transmit

and receive the maximum stresses produced by the test without lost motion, vibration, or sudden shock.

Three-Edge Bearings.

20. (See Fig. 3.)—When three-edge bearings are used, the ends of each specimen of tile shall be accurately marked in halves of the circumference prior to the test.

The lower bearings shall consist of two wooden strips with vertical sides, each strip having its interior top corner rounded to a radius of approximately $\frac{1}{2}$ in. They shall be straight, and shall be securely fastened to a rigid block with their interior vertical sides 1 in. apart.

The upper bearing shall be a wooden block, straight and true from end to end.

The test load shall be applied through the upper bearing block in such a way as to leave the bearing free to move in a vertical plane passing midway between the lower bearings.

In testing a tile which is "out of straight," the lines of the bearings chosen shall be from those which appear to give most favorable conditions for fair bearings.

(*B*) *Absorption Tests of Drain Tile.*

Test Specimens.

21. Not less than three separate test specimens from each of five separate tiles shall be taken as a "standard sample" for the absorption test. Of the three specimens from each tile, one shall be taken from one end, another from the opposite end, and the third shall be taken from the middle portion of the tile. Each specimen shall be of from 12 to 20 sq. in. in area, measured upon the exterior or convex side, and shall be as nearly square as the nature of the material will readily permit. The specimens shall be obtained by breaking the tile, and shall be apparently sound, solid pieces of the wall of the tile, and shall not show cracks or fissures or shattered edges due to the shock of breaking or cutting. The specimens may be obtained from the broken pieces of the tiles used in the strength test, if the restrictions as to the size and location of the specimens can be duly observed. The specimens shall be so marked as to permit the identity of each one to be ascertained at any stage of the test.

Drying Test Specimens.

22. Preparatory to the absorption test, all specimens shall be first weighed and then dried in a drier or oven, at a temperature of not less than 110° C. (230° F.) for not less than three hours. After removal from the drier, the specimens shall be

allowed to cool to a temperature of 20 to 25° C. (68 to 77° F.) and reweighed. If the specimens were apparently dry when taken, and the second weight closely checks the first, the specimens shall be considered dry. If the specimens were known to be wet when taken, they shall be placed in the drier for a further drying treatment of two hours, and reweighed. If the third weight checks the second, the specimens shall be considered dry. In case of any doubt, the specimens must be redried for two-hour periods until check weights are obtained.

Weighing and Reweighing.

23. The balance used shall be sensitive to 0.5 g. when loaded with 1 kg., and weighings shall be read at least to the nearest gram. Where other than metric weights are used, the same order of accuracy must be obtained.

In reweighing after immersion, the specimens shall be removed from the water, not allowed to drain for more than one minute, the superficial water removed by towel or blotting paper, and the specimens at once put upon the balance.

Immersion of Test Specimens.

24. Specimens after weighing shall be placed in a suitable woven-wire receptacle, packed tightly enough to prevent jostling, covered with distilled water or rainwater, raised to the boiling point and boiled for five hours, and then cooled in water to a final temperature of 10 to 15° C. (50 to 59° F.).

Calculation and Reporting of Results.

25. The test results shall be calculated as percentages of the initial dry weight, carried to the nearest first decimal place. The results shall be reported separately for each individual specimen, together with the mean of the fifteen or more specimens comprising the standard sample, the maximum and the minimum single observations entering into the mean, and the variation between the maximum and the minimum of the three specimens of each tile represented in the standard sample.

(*C*) *Freezing and Thawing Tests of Drain Tile.*

Test Specimens.

26. The test specimens employed in making the freezing and thawing test shall be another set selected as specified in Section 21.

Drying Test Specimens.

27. The specimens shall be dried as specified in Section 22.

Weighing and Reweighing.

28. The same scales and weights as are specified in Section 23 for the absorption test or others of equivalent sensitiveness and accuracy shall be employed for the weighings required in the freezing and thawing test. The same procedure in weighings and reweighing as specified in Section 23 shall be used.

Immersion of Test Specimens.

29. The specimens shall be immersed for 72 hours in water having a temperature of 18 to 24° C. (65 to 75° F.) and then weighed as specified in Section 23.

Freezing and Thawing.

30. When the specimens (either from the absorption test or from a specially prepared series) have been weighed after saturation with water, they shall be returned to the water, and kept immersed till the freezing test is begun. For freezing, they shall be placed with their concave faces upward in watertight metal trays, suitably mounted in a rigid metal crate,[1] and immersed in ice water until the specimens have attained substantially the temperature of the water, after which the water shall be drawn down to a depth of $\frac{1}{2}$ in. in each tray. The crate shall then be lifted as a whole, without disturbing the specimens, and placed in the freezing apparatus.

Freezing shall be performed in a quiet atmosphere, free from perceptible natural or artificial currents. If artificial freezing apparatus is employed,[2] the apparatus shall have sufficient heat-absorbent capacity to enable the temperature of the freezing chamber to be brought to −10° C. (+14° F.) or below, within thirty minutes after the introduction of the specimens. The temperature in the freezing apparatus shall not fall lower than −20° C. (−4° F.). The freezing shall be continued until the water in the trays is frozen solid. Exposure to freezing conditions in excess of this requirement shall be considered as without significance.

At the conclusion of freezing under the specified conditions, the crate of specimens shall be withdrawn and at once immersed in water at a temperature of 18 to 24° C. (65 to 75° F.) in a special receptacle of proper size. Heating shall be continued so that the water will regain the required temperature as soon as practicable after the specimens are immersed. A temperature of 18 to 24° C. (65 to 75° F.) shall then be maintained for not less than 2 hours. At the conclusion of the thawing treatment, the crate of specimens shall be inspected and the condition of each sample after each thawing shall be noted in the records.

Method of Determining Failure in Freezing and Thawing Tests.

31. Failure under the freezing and thawing treatment shall be considered to be reached when:

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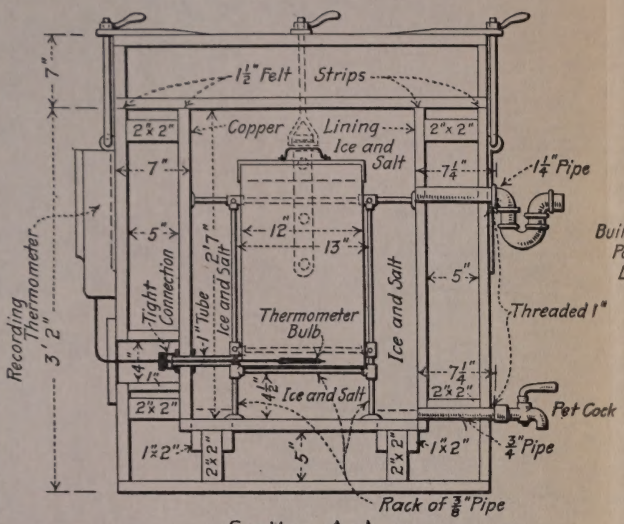
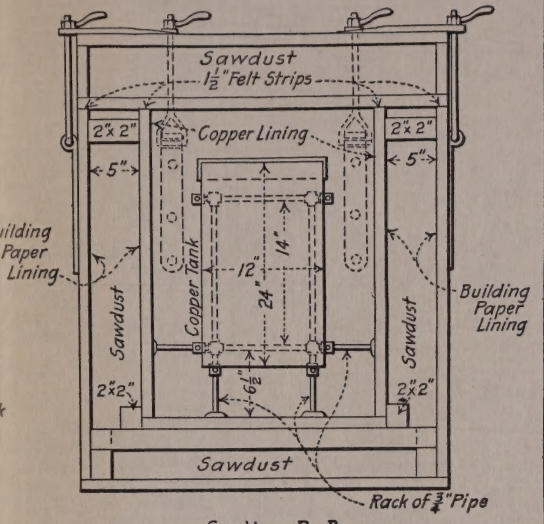
[1] Fig. 4 shows a crate and trays suitable for use in the box for artificial freezing illustrated in Plate IV.

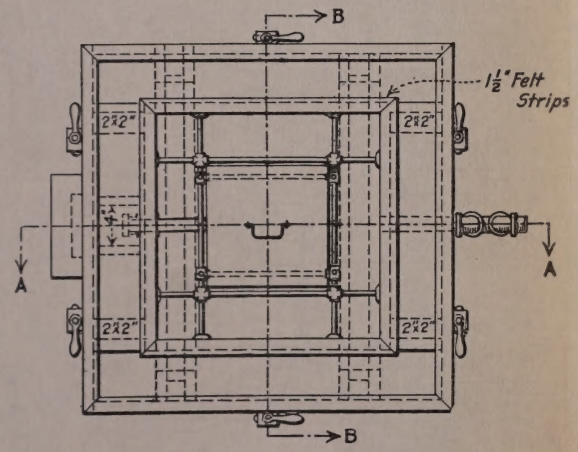
[2] Artificial freezing will generally be necessary. It may be conducted in a commercial zero (F.) refrigerating room, or in an artificial freezing box similar to the one shown in Plate IV, in which zero (F.) temperatures can readily be produced by the use of salt and ice.



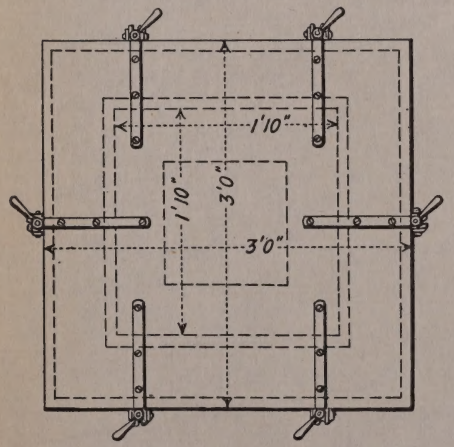
Section A-A.



Section B-B



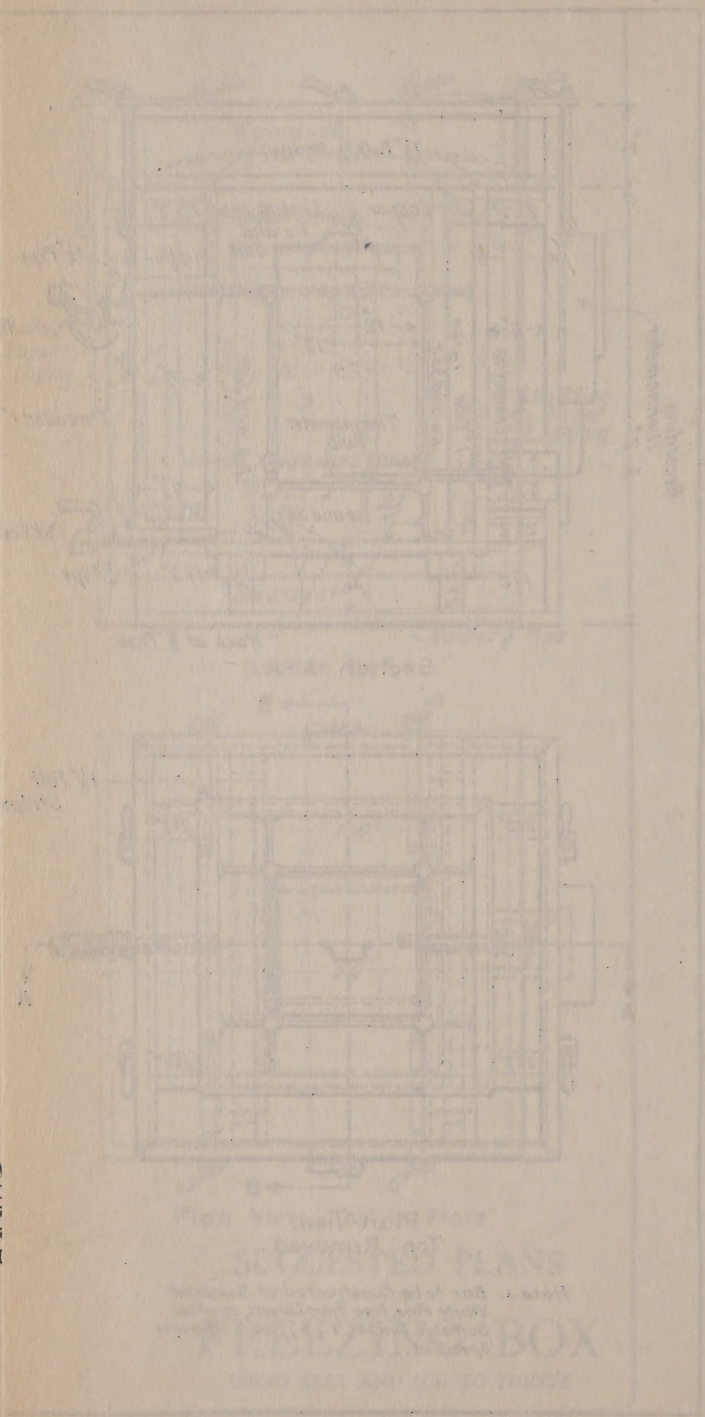
Plan View,
Top Removed.



Plan View, Top in Place

Note: Box to be Constructed of Seasoned White Pine, free from Defects, or other Suitable Timber, 1"x 6", unless otherwise Specified.

SUGGESTED PLANS
FOR
FREEZING BOX
USING SALT AND ICE TO FREEZE



(*a*) The specimens show superficial disintegration or spalling with loss of weight of more than 5 per cent of the initial dry weight; or,

(*b*) The specimens are badly cracked in other than lamination planes; or,

(*c*) The specimens show evident serious loss of structural strength.

PHYSICAL TEST REQUIREMENTS

Physical Test Requirements.

32. The physical test requirements for the different classes of drain tile shall be as given in Table I.

Absorption Requirements for Drain Tile made of Mixed Clays.

33. Drain tile made of mixtures of surface clays with other clays shall conform to the absorption requirements for surface-clay tile in Table I, when the proportion of surface clay is 75 per cent or more, and to the requirements for shale and fire-clay tile for all other proportions.

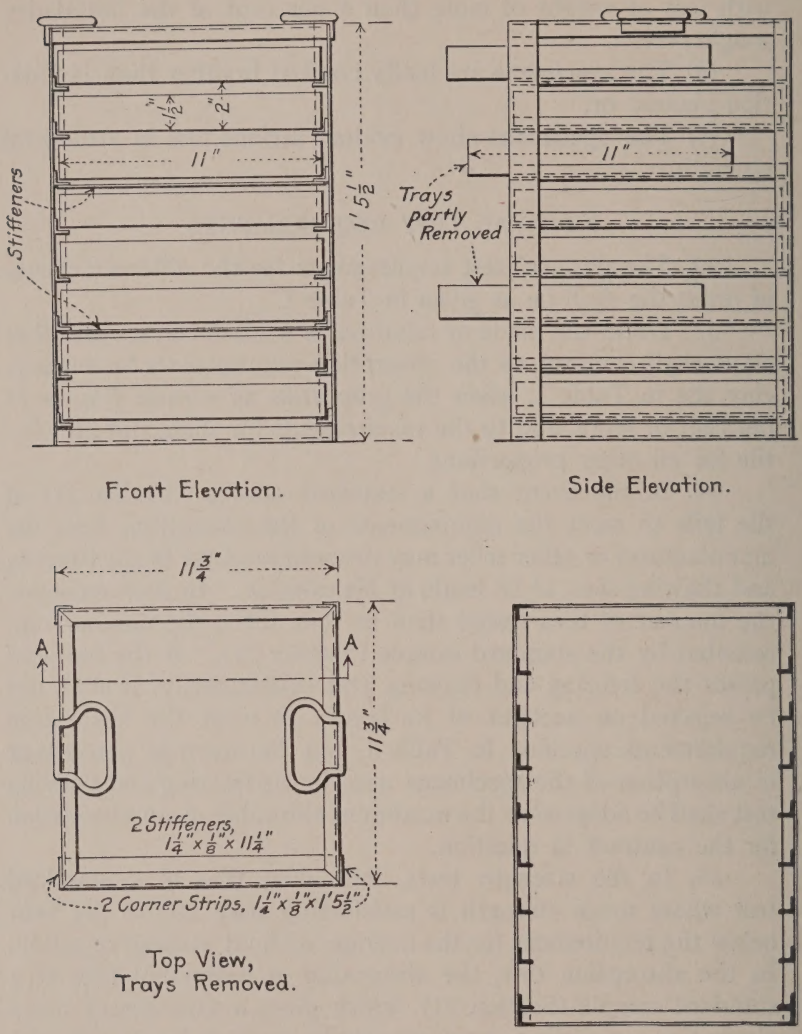
Appeal from Absorption Test to Freezing and Thawing Test.

34. In the event that a standard sample (Section 21) of tile fails to meet the requirements of the absorption test, the manufacturer or other seller may demand recourse to the freezing and thawing test, to be made at his expense. In such recourse, the number of tiles tested shall be four times the number represented by the standard sample (Section 21). If the material passes the freezing and thawing test satisfactorily, it shall not be rejected on account of its failure to meet the absorption requirements specified in Table I, but the average percentage of absorption of the specimens used in the freezing and thawing test shall be adopted as the maximum allowable mean absorption for the contract in question.

Limits of Fluctuation of Individual Test Specimens in Physical Tests; Culling and Retesting when Limits Exceeded.

35. In the strength tests, individual tiles of a standard test whose mean strength is satisfactory may fall 25 per cent below the requirement for the average without causing rejection. In the absorption test, the absorption of individual tiles of a standard sample (Section 21), which gives a satisfactory mean absorption percentage, may exceed the average by 25 per cent without causing rejection. In the freezing and thawing test, at least 95 per cent of all the tiles tested shall meet the requirement.

In the event of the failure of a standard sample (Sections 9, 21 and 26) to meet the above requirements, the manufacturer or other seller may thoroughly cull the material and submit a portion for retest at his own expense, and for such retest the number of tiles per sample shall be 10 for the strength and



Notes:

Rack Constructed of $\frac{5}{8}$" x $\frac{5}{8}$" x $\frac{1}{8}$" Galvanized Angles, except as Noted.
All Connections Riveted or Soldered.
Trays, 11" x 11" x 1½" outside, Made of No. 17 Galvanized Steel.

FIG. 4.—Suggested Plans for Freezing Crate and Trays.

absorption tests and 20 for the freezing and thawing test. In the event of the failure of the material after culling to pass the requirements, it shall be rejected without further test.

TABLE I.—PHYSICAL TEST REQUIREMENTS FOR DIFFERENT CLASSES OF DRAIN TILE.

CAUTION.—Drain tile meeting these requirements are not necessarily safe against cracking in ditches. For safe requirements as against cracking, see Table II.

Internal Diameter of Tile, in.	Farm Drain Tile				Standard Drain Tile				Extra-Quality Drain Tile			
	Minimum Average Ordinary Supporting Strength, lb. per linear ft.	Maximum Average Absorption by Standard Boiling Test, per cent			Minimum Average Ordinary Supporting Strength, lb. per linear ft.	Maximum Average Absorption by Standard Boiling Test, per cent			Minimum Average Ordinary Supporting Strength, lb. per linear ft.	Maximum Average Absorption by Standard Boiling Test, per cent		
		Shale and Fire-Clay Tile	Surface-Clay Tile	Concrete Tile		Shale and Fire-Clay Tile	Surface-Clay Tile	Concrete Tile		Shale and Fire-Clay Tile	Surface-Clay Tile	Concrete Tile
4.....	800	11	14	12	1200	9	13	10	1600	7	11	9
5.....	800	11	14	12	1200	9	13	10	1600	7	11	9
6.....	800	11	14	12	1200	9	13	10	1600	7	11	9
8.....	800	11	14	12	1200	9	13	10	1600	7	11	9
10.....	800	11	14	12	1200	9	13	10	1600	7	11	9
12.....	800	11	14	12	1200	9	13	10	1600	7	11	9
15.....	1000	11	14	12	1300	9	13	10	1600	7	11	9
18.....					1400	9	13	10	1800	7	11	9
21.....					1550	9	13	10	2100	7	11	9
24.....	(Not Permitted)	(Not Permitted)	(Not Permitted)	(Not Permitted)	1700	9	13	10	2400	7	11	9
27.....					1850	9	13	10	2700	7	11	9
30.....					2000	9	13	10	3000	7	11	9
33.....					2150	9	13	10	3300	7	11	9
36.....					2300	9	13	10	3600	7	11	9
39.....					2450	9	13	10	3900	7	11	9
42[a].....					2600	9	13	10	4200	7	11	9

[a] Larger sizes recommended as standard are as follows: 45, 48, 54, and 60 in.

NOTE.—When the freezing and thawing test is made as provided in Section 7, the number of freezings and thawings to be endured shall be as follows: For farm drain tile, 24; for standard drain tile, 36; for extra-quality drain tile, 48.

Strength Test Requirements when Manufacturer is held Responsible for Cracking in Ditches.

36. The manufacturer or other seller shall not be held responsible for cracking of drain tile in ditches unless by special agreement in advance, and in any event his obligation shall be held to be discharged by the delivery of drain tile having the minimum ordinary supporting strengths specified in Table II; and, if it is not otherwise specified in advance by the purchaser, tile shall be supplied of the strengths specified for clay ditch filling, for "ordinary" pipe laying and for widths of ditch at the level of the top of the tile equal to 0.5 ft. greater than the outside diameters of the tile. The purchaser shall furnish to the manufacturer or other seller complete information, in advance of

TABLE II.—STANDARD ORDINARY SUPPORTING STRENGTHS OF DRAIN TILE FOR ORDINARY SAND AND FOR THOROUGHLY WET CLAY DITCH FILLING MATERIALS.

STRENGTHS IN POUNDS PER LINEAR FOOT.

Height of Fill above Top of Tile, ft.	Breadth of Ditch at Top of Tile.																			
	1 ft.				2 ft.				3 ft.				4 ft.				5 ft.			
	Method of Laying Pipe.				Method of Laying Pipe.				Method of Laying Pipe.				Method of Laying Pipe.				Method of Laying Pipe.			
	Ordinary.		First Class.		Ordinary.		First Class.		Ordinary.		First Class.		Ordinary.		First Class.		Ordinary.		First Class.	
	Ditch Filling Material.		Ditch Filling Material.		Ditch Filling Material.		Ditch Filling Material.		Ditch Filling Material.		Ditch Filling Material.		Ditch Filling Material.		Ditch Filling Material.		Ditch Filling Material.		Ditch Filling Material.	
	Sand.	Clay.	Sand.	Clay.	Sand.	Clay.	Sand.	Clay.	Sand.	Clay.	Sand.	Clay.	Sand.	Clay.	Sand.	Clay.	Sand.	Clay.	Sand.	Clay.
2	265	280	220	235	615	635	510	530	970	990	810	830	1 330	1 350	1 110	1 130	1 690	1 710	1 410	1 430
4	400	450	335	375	1 050	1 120	880	935	1 750	1 820	1 460	1 520	2 450	2 540	2 040	2 110	3 160	3 250	2 640	2 710
6	470	545	390	455	1 370	1 500	1 140	1 250	2 370	2 530	1 980	2 110	3 410	3 580	2 840	2 980	4 460	4 640	3 720	3 870
8	505	605	420	505	1 600	1 790	1 330	1 490	2 870	3 110	2 390	2 590	4 220	4 490	3 510	3 740	5 590	5 890	4 660	4 910
10	525	640	440	535	1 760	2 010	1 470	1 680	3 270	3 610	2 730	3 010	4 900	5 290	4 080	4 410	6 590	7 020	5 490	5 850
12	535	660	445	550	1 880	2 190	1 570	1 820	3 600	4 030	3 000	3 355	5 480	6 000	4 570	5 000	7 460	8 030	6 220	6 690
14	540	675	450	560	1 960	2 320	1 640	1 930	3 850	4 380	3 210	3 650	5 980	6 620	4 980	5 520	8 230	8 950	6 850	7 460
16	545	680	455	565	2 030	2 420	1 690	2 020	4 060	4 670	3 390	3 890	6 400	7 160	5 330	5 970	8 890	9 770	7 410	8 150
18	545	685	455	570	2 070	2 500	1 720	2 090	4 230	4 920	3 530	4 100	6 750	7 630	5 630	6 360	9 480	10 520	7 900	8 770
20	545	690	455	575	2 100	2 560	1 750	2 140	4 370	5 130	3 640	4 280	7 050	8 060	5 880	6 720	9 990	11 200	8 330	9 330
22	545	690	455	575	2 120	2 610	1 770	2 180	4 470	5 310	3 730	4 420	7 310	8 430	6 090	7 020	10 400	11 800	8 700	9 830
24	545	690	455	575	2 140	2 650	1 780	2 210	4 560	5 450	3 800	4 540	7 520	8 750	6 270	7 290	10 800	12 300	9 030	10 300
26	545	690	455	575	2 150	2 670	1 790	2 230	4 630	5 580	3 850	4 640	7 700	9 030	6 420	7 530	11 200	12 800	9 320	10 700
28	545	690	455	575	2 160	2 700	1 800	2 250	4 680	5 680	3 900	4 740	7 860	9 280	6 550	7 740	11 500	13 300	9 570	11 100
30	545	690	455	575	2 170	2 710	1 810	2 260	4 730	5 770	3 940	4 810	7 990	9 500	6 660	7 920	11 800	13 700	9 800	11 400
Very great...	545	690	455	575	2 180	2 770	1 820	2 310	4 910	6 230	4 090	5 190	8 730	11 100	7 270	9 230	13 600	17 300	11 400	14 400

NOTE.—Ordinary Pipe Laying is pipe laying in accordance with customary good practice in tile-drain construction, whereby the underside of the pipe is well bedded on soil for 60 to 90 deg. of the circumference.

First-Class Pipe Laying is pipe laying in accordance with the best customary practice in pipe-sewer construction, whereby the entire underside of the pipe is very thoroughly bedded on soil and the entire pipe is surrounded by well-compacted soil, under the direction of an inspector constantly present on the work.

When pipe is laid in a Concrete or Other Permanent Masonry Cradle, strong enough to carry the entire load to the sub-base without breaking and large enough to prevent material settlement, the standard strengths for all dimensions of ditches and all filling materials shall be those specified for Standard Drain Tile in Table I.

receiving bids, as to the number of linear feet of drain tile of each diameter required for each different depth of ditch, measured to the nearest foot from the surface of the ground to the top of the tile.

VISUAL INSPECTION

Visual Inspection and its Purposes.

37. All drain tile shall be given a thorough visual inspection at the trench by a competent inspector employed by the purchaser. The purposes of the visual inspection shall be: (1) to cull and reject imperfect individual tiles; and (2) to determine whether the tiles, independently of meeting the chemical and the physical test requirements, comply with the specifications of general properties, especially as stated hereinafter.

Shape.

38. All drain tile shall be of approximately circular cross-section, except when otherwise specified in advance. They shall be approximately straight, except in the case of special connections. The ends shall be so regular and smooth as readily to admit of making close joints by turning and pressing together adjoining tile.

Nominal Sizes.

39. The sizes of drain tile shall be designated by their interior diameters.

Minimum Lengths.

40. Drain tile smaller than 12 in. in diameter shall have a minimum length of 12 in. Tile of from 12 to 30 in. in diameter, inclusive, shall have lengths not less than the diameters. Tile larger than 30 in. in diameter shall have a minimum length of 30 in.

Structure.

41. Drain tile shall be substantially uniform in structure throughout, and the inspector shall investigate this property by examining fractured surfaces.

Ring.

42. Drain tile shall give a clear ring when stood on end and while dry tapped with a light hammer.

Color.

43. The inspector may use the color of drain tile as a general guide in sorting and inspecting, but he shall first so familiarize himself with the raw materials and the processes used in the manufacture of the particular tile in question as to be competent to interpret the true meaning of variations in their color.

Inside Smoothness.

44. Drain tile shall be reasonably smooth on the inside.

Cracks, Checks, Chips and Broken Pieces.

45. Drain tile shall be free from cracks and checks extending into the body of the tile in such a manner as to decrease the strength appreciably. Tile shall not be chipped or broken in such a manner as to decrease their strength materially or to admit earth into the drain.

Use of the Terms Vitrified and Hard Burned.

46. All drain tile shall be sufficiently "vitrified" or "hard-burned" to afford the degree of supporting strength, percentage of absorption, and resistance to freezing and thawing specified in the physical test requirements prescribed in Table I.

Appeal from Results of Visual Inspection.

47. The manufacturer or other seller may appeal from decisions of the inspector on questions of strength or structure when such decisions are based on visual inspection alone, in which case the point at issue shall be determined by standard physical tests, the cost of which shall be paid by the appellant, if the inspector was right, or by the purchaser if his inspector was in error.

TABLE III.—DISTINCTIVE GENERAL PHYSICAL PROPERTIES OF DIFFERENT CLASSES OF DRAIN TILE.

Physical Properties Specified.	Farm Drain Tile.	Standard Drain Tile.	Extra-Quality Drain Tile.
Allowable variation of average diameter below specified diameter, per cent	5	3	3
Allowable variation between maximum and minimum diameters of same tile, or average diameters of adjoining tile, percentage of thickness of wall	85	75	65
Allowable variation from straightness, percentage of length.	5	3	3
Allowable thickness of exterior blisters, lumps and flakes which do not weaken tile and are few in number, percentage of thickness of wall	25	20	15
Allowable diameters of above blisters, lumps and flakes, percentage of internal diameter	20	15	10
General Inspection	Careful.	Rigid.	Very rigid.

Additional Distinctive Physical Characteristics.

48. Drain tile of the different classes shall, in addition to all requirements heretofore specified, have the distinctive physical characteristic prescribed in Table III.

TESTING, INSPECTION AND REJECTION

Making and Reporting Tests.

49. All tests shall be made by experts employed by the purchaser. Full reports of all tests shall be furnished the manufacturer or other seller on his request. Tests shall be made and reported promptly.

Expense of Making Tests.

50. The purchaser shall pay the expense of making all tests except as otherwise specified in Sections 9, 34, 35, 47 and 52.

Number of Tests.

51. The number of standard tests to be made shall be determined by the purchaser.

General Tests and Inspection at Factory.

52. In all contracts for ten or more carloads of tile, preliminary general tests and inspection shall be made at the

factory by the purchaser upon demand of the manufacturer or other seller. The expense of such tests and inspection shall be paid by the manufacturer or other seller.

Inspector.

53. The inspector shall be employed by the purchaser.

Inspection.

54. The manufacturer or other seller of the drain tile shall afford the inspector all reasonable facilities for his work, both as to the selection of specimens for tests, and as to visual inspection. Inspection shall be completed promptly.

Rejection.

55. The inspector shall plainly mark all drain tile which he rejects, and such rejected tile shall be removed promptly by the manufacturer or other seller. Upon request of the purchaser, the manufacturer or other seller shall give full account of the removal of rejected tile.

STANDARD DEFINITIONS
OF
TERMS RELATING TO SEWER PIPE[1]

Serial Designation: C 8 – 24

These definitions are issued under the fixed designation C 8; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1915; REVISED, 1924.

FORCES ACTING UPON SEWER PIPE

External Forces.—Forces resulting from pressures or impact acting from the outside upon the pipe.

Internal Forces.—Forces resulting from interior fluid pressure.

Abrasion.—Wearing away of surface material by friction.

RAW MATERIALS

Physical Properties.—Those sensible properties of raw materials, which in their combinations affect the manufacture and use of the product.

Chemical Properties.—Those properties resulting from combinations of elements in the raw materials, which in their composition affect the manufacture and use of the product.

Clay.—An earthy or stony mineral aggregate consisting essentially of hydrous silicates of alumina, plastic when sufficiently pulverized and wetted, rigid when dry and vitreous when burned at a sufficiently high temperature.

Surface Clay.—An unconsolidated, unstratified clay, occurring on the surface.

Fire Clay.—A sedimentary clay of low flux content.

NOTE.—It is usually associated with coal measures.

Shale.—A thinly stratified, consolidated sedimentary clay with well-marked cleavage parallel to the bedding.

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee C-4 on Clay and Cement-Concrete Pipe.

PIPE

(*a*) *Sewer Pipe.*—Separate pipe suitable for the conveyance of domestic and industrial sewage and storm water and capable of being properly joined to form a conduit.

Clay Pipe.—Made from red burning plastic clay devoid of fissile structure. Maturing temperature about 1170° C. Vitrification not ordinarily produced, and salt glazing not always effective.

Fire-Clay Pipe.—Made from buff, gray or reddish burning fire clay showing conchoidal structure. Maturing temperature about 1250° C. Complete stony vitrification may be produced, but an absorption lower than 3 per cent is not general, nor desirable for maximum strength.

Shale Pipe.—Made from red burning hard clay with a distinct fissile structure. Vitrification at from 1050 to 1250° C., and salt glazing successful only at highest temperature.

Cement-Concrete Pipe.—Pipe formed by consolidating in a mold a mixture of Portland cement, water, sand, stone or other hard material, and permitting it to harden by natural process prior to handling and use.

Special.—A pipe other than a straight pipe.

Branch.—A pipe attached to and diverging from the barrel of another pipe, such as Y-branches, T-branches, etc.

(*b*) *Demands upon Sewer Pipes.*—Requirements of qualities which are desirable and attainable under conditions of actual practice.

PARTS OF SEWER PIPE

Barrel or Shell.—Main body of a pipe, exclusive of differently formed ends.

Ends.—Those parts of a pipe which terminate it and are so formed as to permit of making a proper joint.

Beveled End.—End surfaces of pipes inclined at an angle with the pipe axis so formed that the end of one pipe can enter the end of the adjoining pipe with a close fit.

Hub or Bell.—That end of a pipe which is sufficiently enlarged for a short distance to receive and enclose the spigot end of the adjoining pipe, to form a joint.

Spigot.—That end of a pipe which enters and is formed to fit the hub or bell of the adjoining pipe; it is sometimes scored or has a head.

Butt.—Plain end of a pipe sometimes scored on the outside, to close up against a similar end of an adjoining pipe for the purpose of forming a joint, the two ends being surrounded and covered by a collar.

Collar.—A flat band to surround and cover a butt joint.

Base, form of.—Shape of that part of a pipe which rests upon a foundation.

JOINTS

Beveled.—Formed by joining pipes with beveled ends and applying the jointing material.

Hub and Spigot.—Formed by inserting the spigot end of one pipe into the hub of another and applying the jointing material.

Butt and Collar.—Formed by abutting the butt ends of two adjacent pipes and, after applying the jointing material, surrounding the joint with a collar.

Jointing Materials.—The materials which are inserted between the ends of adjoining pipes for the purpose of forming a continuous closed conduit.

Elasticity of Joints.—Ability of jointed pipes to resist strains caused by bending and returning to original position.

FINISHED PRODUCT

(*a*) *Material.*

Durability.—Resistance to disintegration or deterioration.

Serviceability.—Ability to readily and effectively render satisfactory service.

Thickness.—Distance between outside and inside surfaces.

Strength.—Ability to resist physical forces imposed during handling and after pipe has been put in use.

Toughness.—Relative degree of resistance to impact without fracture as opposed to brittleness.

Hardness.—Intensity of molecular cohesion as measured by resistance to penetration by a standard tool.

Smoothness.—Evenness of surface without projections or irregularities.

Vitrification.—The consolidation of material under high heat.

Shrinkage.—Diminution of dimensions and mass of the material.
Porosity.—Ratio of the volume of interstices of the material to the volume of its mass.
Percolation.—Passage of water through the interstices of the material.

(*b*) *Covering.*

Coating.—A covering of other materials applied in liquid form.
Glazing.—Hard glassy surface covering.
Salt Glazing.—Glazing produced by application of salt during vitrification.
Lining.—A covering of other material applied in solid form to inside surfaces.
Waterproofing.—Materials resistant to penetration by water.

(*c*) *Defects.*

Warp.—Twisted out of normal shape.
Fracture.—Rupture of the material by a break through its entire thickness.
Crack.—Fissure or open seam not necessarily extending through body of material.
Fire Crack.—A crack resulting from lack of uniformity in shrinkage after the application of excessive heat.
Hair Crack.—Irregularly running, fine, narrow crevice or fissure at the surface of a pipe not penetrating deeply, and caused by a shrinkage of material during manufacture.
Blister.—Convex hollow prominence formed by separation and raising of a film in the process of burning.
Leakage.—Passage of water contrary to intention.
Lamination.—Division of material into thin layers or sheets.

IDENTIFICATION

Marking and Imprints.—Impressions made upon pipe at the place and time of manufacture for the purpose of identification.

RECOMMENDED PRACTICE
FOR
LAYING SEWER PIPE[1]

Serial Designation: C 12 – 19

This recommended practice is issued under the fixed designation C 12; the final number indicates the year of original adoption, or in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1915; ADOPTED IN AMENDED FORM, 1919.

PREPARING TRENCHES AND FOUNDATIONS FOR PIPE LAYING

The foundations in the trench should be formed to prevent any subsequent settlement and thereby possibly an excessive pressure and consequent rupture of the pipes.

If the foundation is rock an equalizing bed of concrete or sand well compacted should be placed upon the rock. The thickness of these beds should be not less than 4 in. Pipes should be laid in these beds so that at least the lower third of each pipe is supported its entire length.

If the foundation is good firm earth, the earth should be pared or molded to give a full support to the lower third of each pipe and, if necessary to secure a proper bearing for the pipe, a layer of concrete, fine gravel or other suitable material should be placed. The same means of securing a firm foundation should be adopted in case the excavation has been made deeper than necessary.

If there is no good natural foundation, the pipes should be laid in a concrete cradle supported on a masonry foundation carried to a soil of satisfactory bearing power or supported on a structure designed to carry the weight of pipe and its load to a firm bearing.

Trenches should be kept free from water until the material in the joints and masonry has sufficiently hardened.

To protect pipe lines from unusual stresses all work should preferably be done in open trenches.

Pipe lines should be placed at a sufficient depth below the surface of the street to avoid dangerous pressure or impact.

[1] Under the standardization procedure of the Society, this recommended practice is under the jurisdiction of the A.S.T.M. Committee C-4 on Clay and Cement-Concrete Pipe.

When this is not possible special reinforcement should be provided.

Trenches should be only of sufficient width to provide a free working space on each side of the pipe, preferably of not over one third of the nominal diameter, and never less than 4 in., according to the size of the pipe and the character of the ground; but in every case there should be sufficient space between the pipe and the sides of the trench to make it possible to thoroughly ram the back-filling around the pipe and to secure tight joints.

PIPE LAYING

The laying of pipes in finished trenches should be commenced at the lowest point, so that the spigot ends point in the direction of flow.

All pipes should be laid with ends abutting and true to line and grade. They should be fitted and matched so that when laid in the work they will form a sewer with a smooth and uniform invert.

It is necessary to use all possible care when shoving the pipes together, so that the joints will not be unnecessarily large.

Sockets should be carefully cleaned before pipes are lowered into trenches. The pipes should be so lowered as to avoid unnecessary handling in the trench.

The pipes should be set firmly according to line and grade, and the joints carefully adjusted and filled with the jointing material.

Joints should be made in the following manner: A closely twisted hemp or oakum gasket of suitable diameter, in no case less than $\frac{3}{4}$ in., and in one piece of sufficient length to pass around the pipe and lap at the top, should be solidly rammed into the annular spaces between the pipes with a suitable calking tool. When cement joints are used, the gasket should first be saturated with neat cement grout. The remainder of the space should then be completely filled with the joining materials.

BACKFILLING TRENCHES

All trenches and excavations should be backfilled immediately after the pipes are laid therein, unless other protection of the pipe line is directed. The backfilling material should be selected and deposited with special reference to the future safety of the pipes. Clean earth, sand or rock dust should be solidly tamped about the pipes up to a level at least 2 ft. above the top

of the pipes. This material should be carefully deposited in uniform layers. Unless otherwise permitted, each layer should be carefully and solidly tamped or rammed with proper tools so as not to injure or disturb the pipe line.

Puddling or water flooding for consolidating the backfilling is recommended only for sandy and gravely materials. If this method is used, the first flooding should be applied after the backfilling has been compacted by tamping up to 2 ft. above the top of the pipes, and the second flooding during or after the subsequent filling of the trench. An excess of water should be avoided, in order to prevent disturbance of the earth under and around the pipes and also to prevent an undue excess of pressure upon them.

Walking or working on the completed sewer, except as may be necessary in tamping or backfilling, should not be permitted until the trench has been backfilled to a height of at least 2 ft. over the top of the pipes.

The filling of the trench should be carried on simultaneously on both sides of the pipes in such a manner that injurious side pressures do not occur.

STANDARD SPECIFICATIONS AND TESTS
FOR
HOLLOW BURNED-CLAY LOAD-BEARING WALL TILE[1]

Serial Designation: C 34 - 27

These specifications are issued under the fixed designation C 34; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1926; REVISED, 1927.

SPECIFICATIONS

Application.

1. These specifications apply to hollow load-bearing wall tile made from surface clay, shale, fire clay or admixtures thereof.

CLASSIFICATION

Classification.

2. (*a*) According to the results of physical tests, tile shall be classified as Hard, Medium, and Soft on the basis of the following strength and absorption requirements, both of which must be met for a given class:[2]

Class	Absorption, per cent		Compressive Strength, Based on Gross Area, lb. per sq. in.[a]			
			End Construction		Side Construction	
	Mean of 5 Tests	Individual Maximum	Mean of 5 Tests	Individual Minimum	Mean of 5 Tests	Individual Minimum
Hard	12 or less	15	2000 or more	1400	1000 or more	700
Medium	16 or less	19	1400 or more	1000	700 or more	500
Soft	25 or less	28	1000 or more	700	500 or more	350

[a] Gross area shall be taken as the total area enclosed by the outside dimensions of the unit taken in a direction perpendicular to that in which the load is carried.

(*b*) Where end-construction tile are used on the side they shall meet the requirements of that construction, and *vice versa*.

(*c*) All tile shall be so designed that substantially the same masonry strength will be developed in all wall thicknesses for which they are to be used.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-10 on Hollow Masonry Building Units.

[2] As different types of clay are used in the manufacture of tile, color cannot be taken as indicative of classification.

WEIGHTS

Weights.

3. (*a*) The tile shall have the following dry weights determined as hereinafter specified:

SIZE OF UNIT, IN.	NUMBER OF CELLS	STANDARD WEIGHT, LB.
3¾ by 12 by 12	3	20
6 by 12 by 12	6	30
8 by 12 by 12	6	36
10 by 12 by 12	6	42
12 by 12 by 12	6	48
12 by 12 by 12	9	52
3¾ by 5 by 12	1	9
8 by 5 by 12	2	16
8 by 5 by 12	3	16
8 by 5 by 12 ("L" Shaped)	..	16
8 by 6½ by 12 ("T" Shaped)	4	16
8 by 7¾ by 12 (Square)	6	24
8 by 10¼ by 12 ("H" Shaped)	7	32
8 by 8 by 8 (Cube)	9	18

(*b*) A tolerance of 5 per cent will be allowed on the above standard weights.

DIMENSIONS

Dimensions.

4. No dimension shall vary more than 3 per cent from the standard dimensions for any form of tile.

WEATHERING RESISTANCE

Weathering Resistance.

5. All tile used in exterior work subjected to weathering conditions shall be able to withstand 100 alternate freezings and thawings. Tile classed as hard or medium by these specifications may be considered as meeting the weathering requirement, provided they are burned to the normal maturity for the given clay. Tile classed as soft shall be accepted as meeting the weathering requirement only on the basis of freezing tests.

FIRE RESISTANCE

Fire Resistance.

6. The tile shall meet the following requirements as tested according to the Tentative Specifications for Fire Tests of Building Construction and Materials (Serial Designation: C 19 – 26 T) of the American Society for Testing Materials[1] as they apply for bearing walls and partitions and to be acceptable shall develop the following resistance periods as tested unplastered:

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I, p. 761 (1926); also 1927 Book of A.S.T.M. Tentative Standards, p. 291.

THICKNESS OF WALL, IN.	NUMBER OF UNITS IN WALL THICKNESS	NUMBER OF CELLS IN WALL	RESISTANCE PERIOD, HOURS[a]
8	1	2	1½
8	1	3	2
12	1	3	3
12	2	3	4
12	2	4	5
16	2 or 3	4	6
16	2 or 3	6	8

[a] These are near the minimum values developed in tests. The average results will generally be higher. Plaster coatings ¾ in. thick applied on both sides and remaining in place throughout the fire test will increase the periods by 1 to 2 hours.

WORKMANSHIP AND FINISH

Workmanship.

7. All tile shall be well burned, reasonably free from laminations and from such cracks, blisters, surface roughness, and other defects as would interfere with the proper setting of the tile or impair the strength or permanence of the construction.

Scoring.

8. The exterior surface of all tile intended for plaster or stucco shall be scored in such a manner as to give good adhesion.

MARKING

Marking.

9. All tile shall bear the word "Load-Bearing" and initials, name or trademark of the manufacturer. These marks shall be indented on the exterior of the tile and shall be plainly legible.

INSPECTION AND REJECTION

Inspection.

10. Proper facilities shall be provided the purchaser for sampling and inspection either at the factory or at the site of the work, as may be specified in the contract. At least 10 days from the time of sampling shall be allowed for the completion of the test. The inspection shall be based on the requirements specified above. All tests shall be made in accordance with the methods hereinafter prescribed.

Rejection.

11. Individual tile shall be rejected for failure to meet the weight, size, or workmanship and finish requirements. In case of failure to meet the absorption and strength requirements for the class specified, the seller may sort the shipment and new samples shall be selected by the purchaser from the retained lot and tested at the expense of the seller. In case the second set of samples fails to meet the test requirements the entire lot shall be rejected.

Expense of Tests.

12. Except as specified in Section 11 and unless otherwise agreed, the expense of inspection and testing shall be borne by the purchaser.

TESTS

SAMPLING

Selection of Samples for Test.

13. Samples of tile for testing shall be selected by the purchaser or by a competent representative authorized by him to do this work. In all cases the samples shall be representative of the whole lot of tile from which they are selected. Full-size tile shall be taken in all cases.

Number of Samples.

14. For the strength, absorption, and weight determinations, five individual tile shall be selected from each kiln or from each 100-ton lot. In no case shall less than five tile be taken. For the fire test the size of the test panel will govern the number of tile required. Samples for the freezing tests shall be taken from tile that have not been subjected to strength or fire tests.

Time and Place of Selecting Samples.

15. Samples for the strength and absorption tests shall be selected at the factory or at the site of the work, as specified. If the fire or freezing tests are to be made it shall be so specified at the time of placing the order, samples for fire tests being selected at the factory at least 45 days in advance of the time of filling the order and 90 days in advance for freezing tests.

Marking Samples.

16. All tile selected for test shall be plainly and permanently marked for reference by the testing operator.

WEIGHT DETERMINATIONS

Weight Determinations.

17. The five tile, if not in kiln-dry condition, shall be dried to constant weight at a temperature of not less than 100° C. (212° F.) and be weighed separately. The scale shall be sensitive to within 0.5 per cent of the weight of the smallest unit.

STRENGTH TESTS

Samples.

18. Five full-size dry tile shall be used.

Speed of Testing Machine.

19. The speed of the moving head of the testing machine shall not be more than 0.05 in. per minute.

Bearing Block.

20. A spherical bearing block of proper design shall be placed on top of the test sample.

Capping.

21. (*a*) Bearing surfaces of the test samples and portions adjoining them which are liable to absorb water from the plastic capping shall be coated with shellac and allowed to dry. A quantity of plastic mortar made of a mixture of three parts (by volume) of portland cement and one part of unretarded gypsum (plaster of Paris) mixed with sufficient water to spread evenly shall be placed on a plain surface which has been coated with oil, and allowed to harden sufficiently

to bear the weight of the tile. The surface to be capped shall be placed on this mortar, and while holding the specimen so that its axis is at right angles to the capping surface it shall be given a single firm pressure.

(*b*) The average thickness of the cap after the extruded plaster has been removed and the edges trued shall not be more than $\frac{1}{8}$ in. Patching of caps after setting shall not be permitted. Imperfect caps shall be removed and replaced with new ones.

(*c*) Where time is not available for aging the cement-gypsum cap, a cap of neat gypsum may be used, although the resulting tile strength will generally be lower than with the cement-gypsum cap. If the tile so capped fail to pass specification requirements on the score of strength, they shall be retested with portland-cement-gypsum caps aged not less than three days.

Time of Testing.

22. When the cement-gypsum cap is used it shall age at least three days before the tile is tested. Where the neat gypsum cap is used the tile may be tested as soon as the plaster has been well set, but not sooner than one hour after the sample has been capped.

Position of Tile.

23. All tile shall be tested in a position such that the load is applied in the same direction as in service.

ABSORPTION TESTS

Selection of Test Samples.

24. The samples shall consist of five tile or three representative pieces from each of these five tile. If small pieces are used, two shall be taken from the shell and one from an interior web, the weight of each piece to be not less than 227 g. ($\frac{1}{2}$ lb.). The samples shall have had their rough edges or loose particles ground off and be free from cracks from the failure of the tile in compression, where taken from tile which have been subjected to strength tests.

Marking Test Samples.

25. Each piece shall be marked so that it may be identified at any time with the tile from which it was taken. Markings which do not cover more than 5 per cent of the total superficial area of the piece shall be used.

Drying the Test Samples.

26. Preparatory to the absorption tests all samples shall be dried to constant weight in a drier or oven at a temperature of not less than 100° C. (212° F.).

Accuracy of Weighings.

27. The balance used shall be sensitive to within 0.2 per cent of the weight of the smallest unit or piece tested.

Saturation of Samples.

28. After obtaining the dry weight of the samples they shall be immersed in soft, distilled or rain water, raised to the boiling point and boiled continuously for one hour, and then allowed to cool in water to room temperature.

Obtaining Saturated Weights.

29. After saturation, the sample shall be removed from the water and allowed to drain for not more than one minute. The superficial water shall be removed with a damp cloth, after which they shall be weighed immediately.

Calculating and Reporting Results.

30. The test results shall be calculated as percentages of the initial dry weight, carried to the nearest first decimal place. The results shall be reported separately for each tile, with the average for the five tile.

FREEZING TESTS

Sampling.

31. Where the freezing test is to be made, five separate representative tile shall be selected.

Preparation of Samples.

32. If not possible to use the whole tile, a piece consisting of a cell not less than 4 in. long shall be sawed from the tile. These pieces shall be saturated by immersion in cold water for at least 72 hours prior to starting the freezing.

Method.

33. Any practical method of obtaining alternate freezings and thawings may be used, the freezings to be always made with fully saturated samples and the time and temperature to be such as to insure full freezing and thawing throughout the specimen. The initial weighing and all weighings for loss shall be made on dry specimens.

Accuracy of Weighings.

34. The balance used shall be sensitive to within 0.1 per cent of the weight of the smallest unit or piece tested.

Interpretation of Results.

35. Failure under the freezing and thawing treatment shall be considered to be reached when:

(*a*) The samples show superficial disintegration or spalling with loss of weight of more than 5 per cent of the initial weight; or

(*b*) The samples are badly cracked; or

(*c*) The samples show evident loss of structural strength.

STANDARD SPECIFICATIONS AND TESTS
FOR
HOLLOW BURNED-CLAY FLOOR TILE[1]

Serial Designation: C 57 – 27

These specifications are issued under the fixed designation C 57; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1924; ADOPTED IN AMENDED FORM, 1927.

SPECIFICATIONS

Application.

1. These specifications apply to hollow floor tile made from surface clay, shale, fire clay or admixtures thereof.

CLASSIFICATION

Classification.

2. (*a*) According to the results of physical tests, tile shall be classified as Hard, Medium, and Soft on the basis of the following strength and absorption requirements, both of which must be met for a given class:[2]

Class	Absorption, per cent		Compressive Strength Based on Net Area, lb. per sq. in.			
			End Construction		Side Construction	
	Mean of 5 Tests	Individual Maximum	Mean of 5 Tests	Individual Minimum	Mean of 5 Tests	Individual Minimum
Hard	12 or less	15	4600 or more	3000	2400 or more	1700
Medium	16 or less	19	3200 or more	2250	1600 or more	1100
Soft	25 or less	28	2000 or more	1400	1200 or more	850

(*b*) Where end-construction tile are used on the side they shall meet the requirements of that construction, and *vice versa.*

WEIGHTS

Weights.

3. (*a*) The tile shall have the following dry weights determined as hereinafter specified:

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-10 on Hollow Masonry Building Units.

[2] As different types of clay are used in the manufacture of tile, color cannot be taken as indicative of classification.

FLAT ARCH

DEPTH OF ARCH, IN.	AVERAGE WEIGHT PER SQUARE FOOT OF FLOOR, LB.
6	26
7	29
8	32
9	35
10	38
12	42
15	50

SEGMENTAL ARCH

DEPTH OF ARCH, IN.	AVERAGE WEIGHT PER SQUARE FOOT OF FLOOR, LB.
6	30
8	36
10	40

TILE FOR USE IN COMBINATION HOLLOW TILE AND CONCRETE CONSTRUCTION

SIZE OF UNIT, IN.	MINIMUM NUMBER OF CELLS	STANDARD WEIGHT, LB.
4 by 12 by 12	3	16
6 by 12 by 12	3	22
6 by 12 by 12	4	25
8 by 12 by 12	4	30
10 by 12 by 12	4	35
12 by 12 by 12	4	40

(*b*) A tolerance of 5 per cent will be allowed on the above standard weights.

DIMENSIONS

Dimensions. 4. No dimension shall vary more than 3 per cent from the standard dimensions for any form of tile.

FIRE RESISTANCE

Fire Resistance. 5. In cases where the fire resistance is an essential property the purchaser shall specify the degree of fire resistance (fire-resistance period) required, and the manufacturer shall supply such available information on the fire test performance of the given or closely similar product as will aid the purchaser in deciding whether the requirements are met. Further tests in accordance with the Tentative Specifications for Fire Tests of Building Construction and Materials (Serial Designation: C 19 – 26 T) of the American Society for Testing Materials[1] may be conducted by the purchaser.

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I, p. 761 (1926); also 1927 Book of A.S.T.M. Tentative Standards, p. 291.

WORKMANSHIP AND FINISH

6. All tile shall be well burned, reasonably free from laminations and from such cracks, blisters, surface roughness and other defects as would interfere with the proper setting of the tile or impair the strength or permanence of the construction. **Workmanship.**

7. The exterior surface of all tile intended for plaster shall be scored in such a manner as to give good adhesion. **Scoring.**

MARKING

8. All tile shall bear the initials, name or trademark of the manufacturer. These marks shall be indented on the exterior of the tile and shall be plainly legible. **Marking.**

INSPECTION AND REJECTION

9. Proper facilities shall be provided the purchaser for sampling and inspection either at the factory or at the site of the work, as may be specified. At least 10 days from the time of sampling shall be allowed for the completion of the test. The inspection shall be based on the requirements specified above. All tests shall be made in accordance with the methods hereinafter prescribed. **Inspection.**

10. (*a*) Individual tile shall be rejected for failure to meet the weight, size, or workmanship and finish requirements. In case of failure to meet the absorption and strength requirements for the class specified, the seller may sort the shipment and new samples shall be selected by the purchaser from the retained lot and tested at the expense of the seller. In case the second set of samples fails to meet the test requirements the entire lot shall be rejected. **Rejection.**

(*b*) By agreement, acceptance may be based on dry weight of the units, percentage absorption, fire resistance and the workmanship and finish. **Acceptance.**

11. Except as specified in Section 10 and unless otherwise agreed, the expense of inspection and testing shall be borne by the purchaser. **Expense of Tests.**

TESTS

SAMPLING

12. Samples of tile for testing shall be selected by the purchaser or by a competent representative authorized by him to do this work. In all cases the samples shall be representative of the whole lot of tile from which they are selected. Full-size tile shall be taken in all cases. **Selection of Samples for Test.**

Number of Samples. 13. For the strength, absorption, and weight determinations, five individual tile shall be selected from each kiln or from each 100-ton lot. Where tile of several designs are used, enough samples shall be weighed to determine the true average weight in pounds per square foot for the construction. In no case shall less than five tile be taken. For the fire test the size of the test panel will govern the number of tile required. Samples for the freezing tests shall be taken from tile that have not been subjected to strength or fire tests.

Time and Place of Selecting Samples. 14. Samples for the strength and absorption tests shall be selected at the factory or at the site of the work, as specified in the contract. If the fire tests are to be made, it shall be so specified at the time of placing the order, samples being selected at the factory at least 45 days in advance of the time of filling the order.

Marking Samples. 15. All tile selected for test shall be plainly and permanently marked for reference by the testing operator.

WEIGHT DETERMINATIONS

Weight Determinations. 16. The five tile, if not in kiln-dry condition, shall be dried to constant weight at a temperature of not less than 100° C. (212° F.) and be weighed separately. The scale shall be sensitive to within 0.5 per cent of the weight of the smallest unit.

STRENGTH TESTS

Samples. 17. Five full-size dry tile shall be used.

Speed of Testing Machine. 18. The speed of the moving head of the testing machine shall not be more than 0.05 in. per minute.

Bearing Block. 19. A spherical bearing block of proper design shall be placed on top of the test sample.

Capping. 20. (*a*) Bearing surfaces of the test samples and portions adjoining them which are liable to absorb water from the plastic capping shall be coated with shellac and allowed to dry. A quantity of plastic mortar made of a mixture of three parts (by volume) of portland cement and one part of unretarded gypsum (plaster of Paris) mixed with sufficient water to spread evenly shall be placed on a plain surface which has been coated with oil, and allowed to harden sufficiently to bear the weight of the tile. The surface to be capped shall be placed on this mortar, and while holding the specimen so that its axis is at right angles to the capping surface it shall be given a single firm pressure.

(*b*) The average thickness of the cap after the extruded plaster has been removed and the edges trued shall not be more than $\frac{1}{8}$ in. Patching of caps after setting shall not be permitted. Imperfect caps shall be removed and replaced with new ones.

(*c*) Where time is not available for aging the cement-gypsum cap, a cap of neat gypsum may be used, although the resulting tile strength will generally be lower than with the cement-gypsum cap. If the tile so capped fail to pass specification requirements on the score of strength, they shall be retested with portland-cement-gypsum caps aged not less than three days.

21. When the cement-gypsum cap is used it shall age at least three days before the tile is tested. Where the neat gypsum cap is used the tile may be tested as soon as the plaster has been well set, but not sooner than one hour after the sample has been capped. **Time of Testing.**

22. All tile shall be tested in a position such that the load is applied in the same direction as in service. **Position of Tile.**

ABSORPTION TESTS

23. The samples shall consist of five tile or three representative pieces from each of these five tile. If small pieces are used two shall be taken from the shell and one from an interior web, the weight of each piece to be not less than 227 g. (½ lb.). The samples shall have had their rough edges or loose particles ground off and be free from cracks from the failure of the tile in compression, where taken from tile which have been subjected to strength tests. **Selection of Test Samples.**

24. Each piece shall be marked so that it may be identified at any time with the tile from which it was taken. Markings which do not cover more than 5 per cent of the total superficial area of the piece shall be used. **Marking Test Samples.**

25. Preparatory to the absorption tests all samples shall be dried to constant weight in a drier or oven at a temperature of not less than 100° C. (212° F.). **Drying the Test Samples.**

26. The balance used shall be sensitive to within 0.2 per cent of the weight of the smallest unit or piece tested. **Accuracy of Weighings.**

27. After obtaining the dry weight of the samples they shall be immersed in soft, distilled or rain water, raised to the boiling point and boiled continuously for one hour, and then allowed to cool in water to room temperature. **Saturation of Samples.**

28. After saturation, the sample shall be removed from the water and allowed to drain for not more than one minute. The superficial water shall be removed with a damp cloth, after which they shall be weighed immediately. **Obtaining Saturated Weights.**

29. The test results shall be calculated as percentages of the initial dry weight, carried to the nearest first decimal place. The results shall be reported separately for each tile, with the average for the five tile. **Calculating and Reporting Results.**

STANDARD DEFINITIONS
OF
TERMS RELATING TO HOLLOW TILE[1]

Serial Designation: C 43 – 24

These definitions are issued under the fixed designation C 43; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1924.

TILE

1. *Hollow Tile.*—Hollow building units with parallel cells.

NOTE.—In the present state of the art the term "hollow tile," if used without a qualifying adjective, is understood to mean clay hollow tile.

The term "terra cotta," which is applied to ornamental building units of burned clay, should not be used to designate hollow tile.

2. *Load-Bearing Wall Tile.*—Hollow tile for use as a load-bearing structural unit in walls.

(*a*) *Hollow Floor Tile.*—Hollow tile for use as a load-bearing structural unit in floors.

(*b*) *Foundation Tile.*—Hollow tile for use as a load-bearing structural unit in foundations.

(*c*) *Side-Construction Tile.*—Hollow tile designed to receive its principal stress at right angles to the direction of its cells.

(*d*) *End-Construction Tile.*—Hollow tile designed to receive its principal stress parallel to the direction of its cells.

(*e*) *Book Tile.*—Hollow tile with tongue and groove edges resembling a book in shape.

(*f*) *Salt-Glazed Tile.*—Clay hollow tile with a vitreous glaze on its surfaces produced by burning salt in the kiln at the temperature used in finishing the burning.

3. *Hollow Tile Fireproofing.*—Hollow tile for use as a protection for structural members against fire.

(*a*) *Split Tile.*—Hollow tile which has been knifed parallel with its cells in the process of manufacture for the purpose of separation into two equal units.

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee C-10 on Hollow Masonry Building Units.

(*b*) *Partition Tile.*—Hollow tile for use in building interior partitions, subdividing areas into rooms or enclosing stairways or shafts, and carrying no superimposed load.

(*c*) *Furring Tile.*—Tile of suitable design for lining the inside of walls and carrying no superimposed load.

(*d*) *Porous Hollow Tile.*—Clay hollow tile in which the natural porosity of the clay has been increased by the admixture of other ingredients.

RAW MATERIALS

4. *Shale.*—A thinly stratified, consolidated sedimentary clay[1] with well-marked cleavage parallel to the bedding.

5. *Fire Clay.*—A sedimentary clay[1] of low flux content.

NOTE.—It is usually associated with coal measures.

6. *Surface Clay.*—An unconsolidated, unstratified clay,[1] occurring on the surface.

DESIGNATION OF DIMENSIONS

7. *Length.*—In the case of hollow tile, that dimension measured between its cut ends.

8. *Thickness.*—In the case of hollow tile, that dimension designed to lie at right angles to the face of the wall, floor, or other member in which it is used.

9. *Width.*—In the case of hollow tile, that dimension measured at right angles to the direction of its thickness and length.

NOTE.—In practice, the first dimension given represents thickness; the second, width; the third, length.

PARTS, OPENINGS AND SURFACE FEATURES

10. *Shell.*—In the case of hollow tile, the outer walls.

11. *Webs.*—In the case of hollow tile, the partitions dividing it into cells.

12. *Cells.*—In the case of hollow tile, the openings parallel with its shell and webs.

13. *Scoring.*—In the case of hollow tile, the grooves formed in the exterior faces of the shell to increase the bond of mortar, plaster, or stucco.

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[1] The definitions for the terms "surface clay," "fire clay," and "shale" are based upon the following definition for the term "clay":

Clay.—An earthy or stony mineral aggregate consisting essentially of hydrous silicates of alumina, plastic when sufficiently pulverized and wetted, rigid when dry and vitreous when burned at a sufficiently high temperature.

STANDARD SPECIFICATIONS

FOR

RAW LINSEED OIL[1]

Serial Designation: D 234 – 27

These specifications are issued under the fixed designation D 234; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1926; ADOPTED IN AMENDED FORM, 1927.

PROPERTIES AND TESTS

Properties.

1. Linseed oil shall be the pure oil pressed from flaxseed and shall conform to the following requirements:

	MAXIMUM	MINIMUM
Specific Gravity 15.5°/15.5° C	0.936	0.931
Acid Number	4.0	.....
Saponification Number	195.0	189.0
Unsaponifiable Matter, per cent	1.50	.....
Iodine Number (Wijs)[a]	.....	177.0
Loss on Heating at 105–110° C., per cent	0.2	.....
Color	Not darker than a freshly prepared solution of 1.0 g. potassium bichromate in 100 cc. pure concentrated sulfuric acid (sp. gr. 1.84).	
Foots, per cent:		
Heated Oil	1.0	.....
Chilled Oil	4.0	.....

[a] If linseed oil of the high-iodine-number type is desired, the minimum iodine number, as specified above, should be changed to 188.0.

METHODS OF TESTING

Methods of Testing.

2. The oil should be tested in accordance with the following methods:

General.—All tests shall be made on oil which has been thoroughly agitated before the removal of a portion for analysis.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

These specifications supersede the Standard Specifications for Purity of Raw Linseed Oil from North American Seed (last issued under the serial designation D 1 – 15) and also supersede the Tentative Specifications for Purity of Raw Linsed Oil from South American Seed (D 77 – 21 T) and the Tentative Specifications for Foots Permissible in Properly Clarified Pure Raw Linseed Oil from North American Seed (D 51 – 18 T).

Specific Gravity.—Use a pyknometer, accurately standardized and having a capacity of at least 25 cc., or any other equally accurate method, making the test at 15.5° C. water being 1.000 at 15.5° C.

Acid Number.—Weigh from 5 to 10 g. of the oil. Transfer to a 300-cc. Erlenmeyer flask. Add 50 cc. of a mixture of equal parts by volume of 95-per-cent ethyl alcohol and c.p. reagent benzol. (This mixture should be previously titrated to a very faint pink with dilute alkali solution, using phenolphthalein as an indicator.) Add phenolphthalein indicator and titrate at once to a faint permanent pink color with 0.2 *N* sodium hydroxide solution. Calculate the acid number (milligrams KOH per gram of oil).

Saponification Number.—Weigh about 2 g. of the oil in a 300-cc. Erlenmeyer flask. Add 25 cc. of alcoholic sodium hydroxide or potassium hydroxide solution. Put a condenser loop inside the neck of the flask and heat on the steam bath for one hour. Cool, add phenolphthalein as indicator, and titrate with 0.5 *N* H_2SO_4. Run two blanks with the alcoholic sodium hydroxide solution. These should check within 0.1 cc. 0.5 *N* H_2SO_4. From the difference between the number of cubic centimeters of 0.5 *N* H_2SO_4 required for the blank and for the determination, calculate the saponification number (milligrams KOH required for 1 g. of the oil).

Unsaponifiable Matter.—Weigh 8 to 10 g. of the oil. Transfer to a 250-cc. long-neck flask. Add 5 cc. of a strong solution of sodium hydroxide (equal weights of NaOH and H_2O) and 50 cc. of 95-per-cent ethyl alcohol. Put a condenser loop inside the neck of the flask and boil for two hours. Occasionally agitate the flask to break up the liquid, but do not project the liquid onto the sides of the flask. At the end of two hours, remove the condenser and allow the liquid to boil down to about 25 cc.

Transfer to a 500-cc. glass-stoppered separatory funnel, rinsing with water. Dilute with water to 250 cc., add 100 cc. of redistilled ether. Stopper and shake for one minute. Let stand until the two layers separate sharp and clear. Draw all but one or two drops of the aqueous layer into a second 500-cc. separatory funnel and repeat the process using 60 cc. of ether. After thorough separation, draw off the aqueous solution into a 400-cc. beaker, then the ether solution into the first separatory funnel, rinsing down with a little water. Return the aqueous solution to the second separatory funnel and shake out again with 60 cc. of ether in a similar manner, finally drawing the aqueous solution into the beaker and rinsing the ether into the first separatory funnel.

Shake the combined ether solution with the combined water rinsings and let the layers separate sharp and clear. Draw off the water and add it to the main aqueous solution. Shake the ether solution with two portions of water (about 25 cc. each). Add these to the main water solution.

Swirl the separatory funnel so as to bring the last drops of water down to the stopcock and draw off until the ether solution just fills the bore of the stopcock. Wipe out the stem of the separatory funnel with a bit of cotton on a wire. Draw the ether solution (portionwise if necessary) into a 250-cc. flask and distill off. While still hot drain the flask into a small weighed beaker, rinsing with a little ether. Evaporate this ether, cool the beaker and weigh. (The unsaponifiable oil from adulterated drying oils may be volatile and as a consequence may evaporate on long heating. Therefore, heat the beaker on a warm plate, occasionally blowing out with a current of dry air. Discontinue heating as soon as the odor of ether is gone.)

Iodine Number.—Place a small quantity of the sample in a small weighing burette or beaker. Weigh accurately. Transfer by dropping from 0.09 to 0.15 g. of oil into a 500-cc. bottle, having a well-ground glass stopper, or an Erlenmeyer flask, having a specially flanged neck for the iodine tests. Reweigh the burette or beaker and determine the amount of sample used. Add 10 cc. of chloroform. Whirl the bottle to dissolve the sample. Add 10 cc. of chloroform to each of two empty bottles like that used for the sample. Add to each bottle 25 cc. of the Wijs solution and let stand with occasional shaking for 1 hour in a dark place at a temperature of from 21 to 23° C. Add 10 cc. of the 15-per-cent potassium iodide solution and 100 cc. of water. Titrate with 0.1 *N* sodium thiosulfate, using starch as an indicator. The titrations on the two blank tests should agree within 0.1 cc. From the difference between the average of the blank titrations and the titration on the samples and the iodine value of the thiosulfate solution calculate the iodine number of the samples tested. (Iodine number is given in centigrams of iodine to 1 g. of sample.)

Preparation of Wijs Iodine Monochloride Solution.—Dissolve iodine in glacial acetic acid that has a melting point of 14.7 to 15° C. and is free from reducing impurities in the proportion so that 13 g. of iodine will be present in 1000 cc. of solution. The preparation of the iodine monochloride solution presents no great difficulty, but it shall be done with care and accuracy in order to obtain satisfactory results. There shall be in the solution no sensible excess either of iodine or more particularly of chlorine over that required to form the monochloride. This condition is most satisfactorily attained by

dissolving in the whole of the acetic acid to be used the requisite quantity of iodine, using a gentle heat to assist the solution, if it is found necessary. Set aside a small portion of this solution while pure, and pass dry chlorine into the remainder until the halogen content of the solution is doubled. Ordinarily it will be found that by passing the chlorine into the main part of the solution until the characteristic color of free iodine has just been discharged, there will be a slight excess of chlorine, which is corrected by the addition of the requisite amount of the unchlorinated portion until all free chlorine has been destroyed. A slight excess of iodine does little or no harm, but excess of chlorine must be avoided.

Loss on Heating at 105 to 110° C.—Place 10 g. of the oil in an accurately weighed 50-cc. Erlenmeyer flask and weigh. Heat in an oven at a temperature between 105 and 110° C. for 30 minutes, then cool and weigh. Calculate the percentage loss. This determination shall be made in a current of carbon dioxide gas.

Color.—Prepare a fresh solution of 1 g. pure potassium bichromate in 100 cc. of pure concentrated colorless sulfuric acid (sp. gr. 1.84). Place the oil and colored concentrated solution in separate thin-walled clear-glass tubes of the same diameter (1 to 2 cm.) to a depth of not less than 25 cm., and compare the depths of color by looking transversely through the columns of liquid by transmitted light.

Determination of Percentage Foots.—Method.—With all materials at a temperature between 20 and 27° C., mix, by shaking for exactly one minute in a graduated tube, 25 cc. of the well-shaken sample of oil, 25 cc. of acetone and 10 cc. of the acid calcium chloride solution. The tube shall then be clamped in an upright position where settling can take place for 24 hours. The temperature during this period should be between 20 and 27° C.

The volume of the stratum lying between the clear calcium chloride solution and the clear acetone and oil mixture is read in 0.1 cc. or a fraction thereof. This reading multiplied by four expresses the amount of foots present as a percentage by volume.

The tube referred to may be a burette or a color comparison tube. It should have an internal diameter of 1.0 to 1.5 cm., and a capacity of not less than 70 cc. The graduations in 0.1 cc. should extend at least from 10 cc. to 50 cc. above the bottom of the tube. The acid calcium chloride solution is prepared by saturating with calcium chloride a mixture of 90 parts water and 10 parts concentrated hydrochloric acid (sp. gr. 1.19).

Heated Oil Test.—Heat a portion of the oil to 65° C., hold it within 2° C. of that temperature for 10 minutes; then cool it to room

temperature (20 to 27° C.). Subject the sample promptly to the foots test as described above.

Chilled Oil Test.—Heat a portion to 65° C., hold it within 2° C. of that temperature for 10 minutes; then place it in a dry clean bottle, stopper tightly, and place in a cracked ice and water mixture (0° C.) for exactly 2 hours. At the end of this time, place the bottle for exactly 30 minutes in a water bath at 25° C., then subject promptly to the foots test as described above.

STANDARD SPECIFICATIONS
FOR
PERILLA OIL, RAW OR REFINED[1]

Serial Designation: D 125 – 23

These specifications are issued under the fixed designation D 125; the final number indicates the year of original adoption as standard, or in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED, 1923.

PROPERTIES AND TESTS

1. Perilla oil, raw or refined, shall conform to the following requirements: **Properties.**

	Maximum	Minimum
Foots, per cent	2.5	...
Loss on heating at 105 to 110° C., per cent	0.2	...
Specific gravity at 15.5°/15.5° C	...	0.932
Acid number	5.0	...
Saponification number	...	190
Iodine number (Hanus)	...	191
Unsaponifiable matter, per cent	1.5	...
Color	Not darker than a freshly prepared solution of 1.0 g. potassium bichromate in 100 cc. pure H_2SO_4 (sp. gr. 1.84).	

METHODS OF TESTING

SOLUTIONS REQUIRED.

2. The following reagents will be required: **Solutions Required.**

Acetone that will pass the specifications of the United States Pharmacopœia.

Acid Calcium Chloride Solution.—Saturate with calcium chloride a mixture of 90 parts water and 10 parts HCl (sp. gr. 1.19).

Standard Sodium Thiosulfate Solution.—Dissolve pure sodium thiosulfate in distilled water that has been well boiled to free it from carbon dioxide in the proportion so that 24.83 g. crystallized sodium thiosulfate will be present in 1000 cc. of the solution. It is best to let this solution stand for about two weeks before standardizing. Standardize with pure resublimed iodine. (See Treadwell-Hall,

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

Analytical Chemistry, Vol. II, third edition, p. 646.) This solution will be approximately 0.1 *N* and it is best to leave it as it is after determining its exact iodine value, rather than to attempt to adjust it to exactly 0.1 *N* strength. Preserve in a stock bottle with a guard tube filled with soda lime.

Starch Solution.—Stir up 2 to 3 g. of potato starch or 5 g. soluble starch with 100 cc. of 1-per-cent salicylic acid solution, add 300 to 400 cc. boiling water, and boil the mixture until the starch is practically dissolved. Dilute to 1 liter.

Potassium Iodide Solution.—Dissolve 150 g. of potassium iodide free from iodate in distilled water and dilute to 1000 cc.

Hanus Solution.—Dissolve 13.2 g. of iodine in 1000 cc. of glacial acetic acid (99.5-per-cent) that will not reduce chromic acid. Add enough bromine to double the halogen content, determined by titration (3 cc. of bromine is about the proper amount). The iodine may be dissolved by the aid of heat, but the solution should be cold when the bromine is added.

Standard Sodium Hydroxide Solution.—Prepare a stock concentrated solution of sodium hydroxide by dissolving NaOH in water in the proportion of 200 g. of NaOH to 200 cc. of water. Allow this solution to cool and settle in a stoppered bottle for several days. Decant the clear liquid from the precipitate of sodium carbonate into another clean bottle. Add clear barium hydroxide solution until no further precipitate forms. Again allow to settle until clear. Draw off about 175 cc. and dilute to 10 liters with freshly boiled distilled water. Preserve in a stock bottle provided with a large guard tube filled with soda lime. Determine the exact strength by titrating against pure benzoic acid (C_6H_5COOH) using phenolphthalein as indicator. (See Bureau of Standards *Scientific Paper No. 183.*) This solution will be approximately 0.25 *N*, but do not attempt to adjust it to any exact value. Determine its exact strength and make proper corrections in using it.

Alcoholic Sodium Hydroxide Solution.—Dissolve pure NaOH in 95-per-cent ethyl alcohol in the proportion of about 22 g. per 1000 cc. Let stand in a stoppered bottle. Decant the clear liquid into another bottle, and keep well stoppered. This solution should be colorless or only slightly yellow when used; it will keep colorless longer if the alcohol is previously treated with NaOH (about 80 g. to 1000 cc.), kept at about 50° C. for 15 days, and then distilled. For an alternate method see *Journal*, American Chemical Society, 1906, p. 395.

Half Normal Sulfuric Acid Solution.—Add about 15 cc. H_2SO_4 (sp. gr. 1.84) to distilled water, cool and dilute to 1000 cc. Determine

the exact strength by titrating against freshly standardized NaOH or by any other accurate method. Either adjust to exactly 0.5 *N* strength or leave as originally made, applying appropriate correction.

METHODS.

3. The oil shall be tested in accordance with the following methods: Methods of Testing.

General.—The laboratory sample shall be thoroughly mixed by shaking, stirring, or pouring from one vessel to another and the samples for the individual tests taken from this thoroughly mixed sample.

Loss on Heating at 105 to 110° C.—Place 10 g. of the oil in an accurately weighed 200-cc. Erlenmeyer flask; weigh. Heat in an oven at a temperature between 105 and 110° C. for 30 minutes; cool and weigh. Calculate the percentage loss. This determination shall be made in a current of dry carbon dioxide gas.

Foots.—With all materials at a temperature between 20 and 27° C., mix, by shaking in a stoppered flask for exactly one minute, 25 cc. of the well-shaken sample of oil, 25 cc. of acetone and 10 cc. of the acid calcium chloride solution. Transfer the mixture to a burette where settling can take place for 24 hours. The temperature during this period should be between 20 and 27° C.

The volume of the stratum lying between the clear calcium chloride solution and the clear acetone and oil mixture is read in tenths of a cubic centimeter or a fraction thereof. This reading multiplied by four expresses the amount of foots present as percentage by volume of the oil taken.

Specific Gravity.—Use a pyknometer accurately standardized and having a capacity of at least 25 cc., or any other equally accurate method, making the test at 15.5° C., water being 1 at 15.5° C.

Acid Number.—Weigh from 5 to 10 g. of the oil. Transfer to a 300-cc. Erlenmeyer flask. Add 50 cc. of neutral 95-per-cent ethyl alcohol. Put a condenser loop inside the neck of the flask. Heat on a steam bath for 30 minutes. Cool and add phenolphthalein indicator. Titrate to a faint permanent pink color with the standard sodium hydroxide solution. Calculate the acid number (milligrams of KOH for 1 g. of the oil).

Saponification Number.—Weigh about 2 g. of the oil in a 300-cc. Erlenmeyer flask. Add 25 cc. of alcoholic sodium hydroxide solution. Put a condenser loop inside the neck of the flask and heat on the steam bath for one hour. Cool, add phenolphthalein as indicator, and titrate with 0.5 *N* H_2SO_4. Run two blanks with the alcoholic sodium hydroxide solution. These should check within 0.1 cc. of

0.5 *N* H_2SO_4. From the difference between the number of cubic centimeters of 0.5 *N* H_2SO_4 required for the blank and for the determination, calculate the saponification number (milligrams of KOH required for 1 g. of the oil).

Unsaponifiable Matter.—Weigh 8 to 10 g. of the oil. Transfer to a 250-cc. long-neck flask. Add 5 cc. of strong solution of sodium hydroxide (equal weights of NaOH and H_2O), and 50 cc. of 95-per-cent ethyl alcohol. Put a condenser loop inside the neck of the flask and boil for two hours. Occasionally agitate the flask to break up the liquid but do not project the liquid onto the sides of the flask. At the end of two hours remove the condenser and allow the liquid to boil down to about 25 cc.

Transfer to a 500-cc. glass-stoppered separatory funnel, rinsing with water. Dilute with water to 250 cc., add 100 cc. of redistilled ether. Stopper and shake for one minute. Let stand until the two layers separate sharp and clear. Draw all but one or two drops of the aqueous layer into a second 500-cc. separatory funnel and repeat the process using 60 cc. of ether. After thorough separation draw off the aqueous solution into a 400-cc. beaker, then the ether solution into the first separatory funnel, rinsing down with a little water. Return the aqueous solution to the second separatory funnel and shake out again with 60 cc. of ether in a similar manner, finally drawing the aqueous solution into the beaker and rinsing the ether into the first separatory funnel.

Shake the combined ether solution with the accumulated water rinsings and let the layers separate sharp and clear. Draw off the water and add it to the main aqueous solution. Shake the ether solution with two portions of water (about 25 cc. each). Add these to the main water solution.

Swirl the separatory funnel so as to bring the last drops of water down to the stop-cock, and draw off until the ether solution just fills the bore of the stop-cock. Wipe out the stem of the separatory funnel with a bit of cotton on a wire. Draw the ether solution (portionwise if necessary) into a 250-cc. flask and distill off. While still hot, drain the flask into a small weighed beaker, rinsing with a little ether. Evaporate this ether, cool and weigh.

NOTE.—The unsaponifiable oil from adulterated drying oils is volatile and will evaporate on long heating. Therefore heat the beaker on a warm plate, occasionally blowing out with a current of dry air. Discontinue heating as soon as the odor of ether is gone.

Hanus Iodine Number.—Place a small quantity of the sample in a small weighing burette or beaker. Weigh accurately. Transfer by

dropping about 0.15 g. (0.10 to 0.20 g.) into a 500-cc. bottle having a well-ground glass stopper, or an Erlenmeyer flask having a specially flanged neck for the iodine test. Reweigh the burette or beaker and determine the amount of sample used. Add 10 cc. of chloroform. Whirl the bottle to dissolve the sample. Add 10 cc. of chloroform to each of two empty bottles like that used for the sample. Add to each bottle 25 cc. of the Hanus solution and let stand with occasional shaking for 30 minutes. Add 10 cc. of the 15-per-cent potassium iodide solution and 100 cc. of water, and titrate with standard sodium thiosulfate using starch as indicator. The titrations on the two blank tests should agree within 0.1 cc. From the difference between the average of the blank titration and the titration on the samples and the iodine value of the thiosulfate solution, calculate the iodine number of the samples tested. (Iodine number is centigrams of iodine to 1 g. of sample.)

Color.—Prepare a fresh solution of pure potassium bichromate in pure colorless H_2SO_4 (sp. gr. 1.84). This solution should be in the proportion of 1.0 g. of potassium bichromate to 100 cc. (184.0 g.) of H_2SO_4. Place the oil and colored solution in separate thin-walled, clear glass tubes of the same diameter (1 to 2 cm.) to a depth of not less than 2.5 cm. and compare the depths of color by looking transversely through the columns of liquid by transmitted light.

STANDARD SPECIFICATIONS
FOR
GUM SPIRITS OF TURPENTINE AND STEAM-DISTILLED WOOD TURPENTINE[1]

Serial Designation: D 13 – 26

These specifications are issued under the fixed designation D 13; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1915; REISSUED AS TENTATIVE, 1920; ADOPTED IN AMENDED FORM, 1924; REVISED, 1926.

Scope.

1. These specifications apply both to spirits of turpentine made from gum (oleoresin) from a living tree, commonly known as "gum spirits" or "turpentine" and to steam-distilled wood turpentine, which is distilled with steam from the oleoresin within the wood. When ordering under these specifications, the purchaser shall specify whether (*a*) gum spirits of turpentine or (*b*) steam-distilled wood turpentine is desired.

PROPERTIES AND TESTS

Properties.

2. Gum spirits of turpentine or steam-distilled wood turpentine shall be pure and conform to the following requirements:

Appearance.

3. It shall be clear and free from suspended matter and water.

Color.

4. The color shall be "Standard" or better.

Odor.

5. The odor shall be mild, aromatic and characteristic of the variety of turpentine specified and, if desired, shall conform to the odor of the sample agreed upon.

Other Properties.

6. Other properties shall be as follows:

	MAXIMUM	MINIMUM
Specific gravity, 15.5°/15.5° C.	0.875	0.860
Refractive index at 20° C.	1.478	1.465
Residue after polymerization with 38 *N* H_2SO_4:		
Volume, per cent	2.0	.....
Refractive index at 20° C.	.....	1.500
Consistency shall be viscous.		
Color shall be straw or darker.		
Initial boiling point at 760 mm. pressure	160° C.	150° C.
Distilling below 170° C. at 760 mm. pressure, per cent	.....	90

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

METHODS OF TESTING

Methods of Testing.

7. The sampling and methods of testing shall be conducted in accordance with the Standard Methods of Sampling and Testing Turpentine (Serial Designation: D 233) of the American Society for Testing Materials.[1]

[1] See p. 269.

STANDARD SPECIFICATIONS
FOR
DESTRUCTIVELY DISTILLED WOOD TURPENTINE[1]

Serial Designation: D 236 – 27

These specifications are issued under the fixed designation D 236; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1926; ADOPTED, 1927.

Scope.

1. These specifications apply only to destructively-distilled wood turpentine obtained in the destructive distillation of resinous wood.

PROPERTIES AND TESTS

Properties.

2. The destructively distilled wood turpentine shall be pure and conform to the following requirements:

Appearance.

3. It shall be clear and free from suspended matter and water.

Color.

4. The color shall be "Standard" or better.

Odor.

5. The odor shall be characteristic of destructively-distilled wood turpentine and, if desired, shall conform to the odor of the sample agreed upon.

Other Properties.

6. Other properties shall be as follows:

	MAXIMUM	MINIMUM
Specific gravity, 15.5°/15.5° C.	0.875	0.860
Refractive index at 20° C, *D* line	1.483	1.463
Residue after polymerization with 38 *N* H_2SO_4:		
Volume, per cent	2.0	.....
Refractive index at 20° C	...	1.480
Initial boiling point at 760 mm. pressure	157° C.	150° C.
Distillation below 170° C. at 760 mm. pressure, per cent	...	60
Distilling below 180° C. at 760 mm. pressure, per cent	...	90

METHODS OF TESTING

Methods of Testing.

7. The sampling and methods of testing shall be conducted in accordance with the Standard Methods of Sampling and Testing Turpentine (Serial Designation: D 233) of the American Society for Testing Materials.[2]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

[2] See p. 269.

STANDARD SPECIFICATIONS
FOR
DRY BLEACHED SHELLAC[1]

Serial Designation: D 207 – 26

These specifications are issued under the fixed designation D 207; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1925; ADOPTED, 1926.

1. Dry bleached shellac[2] shall be furnished in two grades, namely, regular and refined.[3] **Grades.**

PROPERTIES AND TESTS

2. Dry bleached shellac shall conform to the following requirements: **Properties.**

	REGULAR	REFINED
Iodine number, maximum	10	10
Matter insoluble in hot alcohol, maximum	1.0 per cent	0.2 per cent
Moisture, maximum	5.0 "	5.0 "
Wax, maximum	5.5 "	0.2 "
Matter soluble in water, maximum	1.0 "	0.3 "
Ash, maximum	1.0 "	0.3 "

METHODS OF TESTING

3. The determination of iodine number, matter insoluble in hot alcohol, moisture, and wax shall be made in accordance with the Standard Methods of Testing Shellac (Serial Designation: D 29) of the American Society for Testing Materials.[4] **Methods of Testing.**

4. The determination of matter soluble in water shall be made as follows: **Matter Soluble in Water.**

Ten to twenty-five grams of dry shellac shall be ground so that 100 per cent shall pass through a No. 20 (840-micron) sieve. The shellac shall be placed in a 250-cc. beaker or flask and 100 cc. of

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

[2] This material is white shellac. It is also known as "bonedry," "vacdry" or "kilndried" bleached shellac.

[3] Refined bleached shellac has been treated in the process of manufacture so as to remove practically all wax and matter insoluble in hot alcohol.

[4] See p. 292.

distilled water added. It shall be stirred thoroughly, covered with a watch glass and permitted to stand at room temperature (21° C.) for four hours, with occasional stirring.

It shall be decanted through a 12.5-cm. filter paper into a weighed evaporating dish, washing the shellac and filter with at least 50 cc. more of water. It shall be evaporated and the extract dried at 105° C. for one hour or more to constant weight. The increase in weight shall be reported as the water-soluble extract.

Ash. 5. The determination of ash shall be made as follows:

Approximately 2 to 3 g. of the sample shall be accurately weighed, transferred to a weighed porcelain or platinum crucible and ignited at as low a temperature as possible until all organic matter has been destroyed. The crucible and contents shall be cooled and weighed and the percentage of ash calculated.

STANDARD SPECIFICATIONS
FOR
ORANGE SHELLAC[1]

Serial Designation: D 237 – 27

These specifications are issued under the fixed designation D 237; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1926; ADOPTED, 1927.

Grades. 1. Orange shellacs may, for convenience, be grouped into the four following grades: Grade A, Grade B, Grade C and Grade D. Stick, seed, garnet and button lacs are not included under these grades.

PROPERTIES AND TESTS

Properties. 2. Orange shellac shall conform to the following requirements:

	GRADE A	GRADE B	GRADE C	GRADE D
Iodine number, maximum	18.0	18.0	18.0	24.3
Matter insoluble in hot 95-per-cent alcohol, maximum per cent	1.75	2.50	3.00	3.00
Moisture and volatile matter, maximum, per cent	2.0	2.0	2.0	2.0
Matter soluble in water, maximum, per cent	0.5	0.5	0.5	0.5
Wax, maximum, per cent	5.5	5.5	5.5	5.5
Ash, maximum, per cent	1.0	1.0	1.0	1.0

SAMPLING

Sampling. 3. (*a*) Only original packages shall be sampled.

(*b*) Samples shall be drawn by hand, or a suitable tryer may be used, preferably from not less than 10 per cent of the lot.

(*c*) In sampling "free" shellac, approximately double handfuls shall be drawn from each bag and quartered down to approximately 2 lb. The shellac shall then be ground to pass through a No. 20 (840-micron) sieve, half of which shall be reserved by the buyer, the other half used for analysis.

(*d*) In sampling "blocky" or "matted" (hard) shellac, about 1 lb. from each bag sampled shall be ground to pass a $\frac{1}{4}$-in. sieve. This shall then be quartered down to approximately a 2-lb. sample which

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

shall be ground to pass through a No. 20 (840-micron) sieve and divided as in sampling "free" shellac.[1]

METHODS OF TESTING

4. The determination of iodine number, matter insoluble in hot alcohol, moisture and wax shall be made in accordance with the Standard Methods of Testing Shellac (Serial Designation: D 29) of the American Society for Testing Materials.[2]

5. The determination of matter soluble in water shall be made as follows:

Weigh 10 to 25 g. of the sample accurately and stir thoroughly with 100 cc. of distilled water in a suitably sized flask or beaker. Cover with a watch glass and allow to stand at room temperature (approximately 21° C.) for four hours, stirring occasionally. Decant the water through a 12.5-cm. filter paper into a weighed evaporating dish, washing the shellac and paper with at least 50 cc. more of water. Evaporate the water and dry the extract at 105 to 110° C. for one hour or more to constant weight. Cool, weigh, and calculate the percentage of matter soluble in water.

6. The determination of ash shall be made as follows:

Approximately 2 to 3 g. of the sample shall be accurately weighed, transferred to a weighed porcelain or platinum crucible and ignited at as low a temperature as possible until all organic matter has been destroyed. The crucible and contents shall be cooled and weighed and the percentage of ash calculated.

EXPLANATORY NOTES

In general, Grade A will include grades of shellac known in the trade as D.C., VSO, Diamond I, Double Triangle G and Superfine.

In general, Grade B will include the grades known in the trade as Fine, Good and Heart Brands.

Grade C represents the grade known in the trade as Pure TN.

Grade D represents the grade known in the trade as U.S.S.A. TN, and which may contain up to 3 per cent of rosin.

[1] The above is a brief abstract of the Rules and Regulations of the United States Shellac Importers Association.

[2] See p. 292.

STANDARD SPECIFICATIONS
FOR
ZINC OXIDE[1]

Serial Designation: D 79 – 24

These specifications are issued under the fixed designation D 79; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1924.

Scope.

1. These specifications cover the pigments commonly known as "Zinc White," or Zinc Oxide, purchased either in the form of dry pigment or ground in oil to form a paste.

MANUFACTURE

Process.

2. (*a*) *Dry Pigment.*—The pigment may be made by the American process direct from the ore, or by the French process from spelter. The order or contract shall state which is desired.

(*b*) *Paste.*—The paste shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) The color and color strength, when specified, shall be equal to that of a sample mutually agreed upon by buyer and seller.

(*b*) *Dry Pigment.*—The pigment shall conform to the following requirements:

	American Process		French Process	
	Maximum	Minimum	Maximum	Minimum
Coarse particles retained on a No. 325 sieve,[2] per cent	1.0	..	1.0	..
Zinc oxide, per cent	...	98	....	99
Total sulfur, per cent	0.2	..	0.1	..
Total impurities, including moisture, per cent	2.0	..	1.0	..

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

[2] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E-11) of the American Society for Testing Materials, see p. 917.

(*c*) *Paste.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency.

The paste shall conform to the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	86	80
Linseed oil, per cent	20	14
Coarse particles and skins (total residue retained on a No. 325 sieve,[1] based on pigment), per cent	1.5	..
Moisture and other volatile matter, per cent	0.5	..

Number of Tests.

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

[1] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

STANDARD SPECIFICATIONS
FOR
LEADED ZINC OXIDE[1]

Serial Designation: D 80 – 24

These specifications are issued under the fixed designation D 80; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1924.

Scope.

1. These specifications cover the pigment commonly known as Leaded Zinc Oxide, and consisting of zinc oxide with varying amounts of lead compounds, purchased either as dry pigment or ground in oil to form a paste.

MANUFACTURE

Process.

2. (*a*) *Dry Pigment.*—The pigment shall be made by the collection of the fumes or dust arising from furnace operations on materials containing zinc.

(*b*) *Paste.*—The paste shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) The color and color strength, when specified, shall be equal to that of a sample mutually agreed upon by buyer and seller.

(*b*) *Dry Pigment.*—The pigment shall conform to the following requirements:

	High-leaded		Low-leaded	
	Maximum	Minimum	Maximum	Minimum
Coarse particles retained on a No. 325 sieve,[2] per cent	1.0	..	1.0	..
Zinc oxide (ZnO), per cent	...	60	...	93
Water-soluble salts, per cent	1.0	..	1.0	..
Total impurities, including moisture, per cent	1.5	..	1.5	..

The remainder shall be normal or basic lead sulfate.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

[2] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E - 11) of the American Society for Testing Materials, see p. 917.

(*c*) *Paste.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency.

The paste shall conform to the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	88.0	83.0
Linseed oil, per cent	17.0	12.0
Moisture and other volatile matter, per cent	0.5	....
Coarse particles and skins (total residue retained on a No. 325 sieve,[1] based on pigment), per cent	1.5	....

Number of Tests.

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

[1] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

STANDARD SPECIFICATIONS
FOR
BASIC CARBONATE WHITE LEAD[1]

Serial Designation: D 81 – 24

These specifications are issued under the fixed designation D 81; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1924.

Scope.

1. These specifications cover what is commonly known as Basic Carbonate White Lead as used as a pigment and in putty, purchased either as dry pigment or ground in oil to form a paste.

MANUFACTURE

Process.

2. (*a*) *Dry Pigment.*—The pigment shall be the product made from metallic lead and shall have a composition corresponding approximately to the formula $2PbCO_3 \cdot Pb(OH)_2$. It shall be thoroughly washed after corroding, shall be free from impurities and adulterants, and shall meet the requirements given in Section 3.

(*b*) *Paste.*—The paste shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) The color and color strength, when specified, shall be equal to that of a sample mutually agreed upon by buyer and seller.

(*b*) *Dry Pigment.*—The pigment shall conform to the following requirements:

	MAXIMUM	MINIMUM
Coarse particles retained on a No. 325 sieve,[2] per cent	1.0	....
Lead carbonate, per cent	75.0	65.0
Total impurities, including moisture, per cent	2.0	....

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

[2] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

(*c*) *Paste.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency.

The paste shall conform to the following requirements:

	MAXIMUM	MINIMUM
Pigment (as specified above), per cent	92	90
Linseed oil, per cent	10	8
Moisture and other volatile matter, per cent	0.7	..
Coarse particles and skins (total residue retained on a No. 325 sieve,[1] based on pigment), per cent	1.5	..

Number of Tests.

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

[1] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

STANDARD SPECIFICATIONS
FOR
BASIC SULFATE WHITE LEAD[1]

Serial Designation: D 82 – 24

These specifications are issued under the fixed designation D 82; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1924.

Scope.

1. These specifications cover the pigment commonly known as "basic sulfate" white lead. The pigment may be purchased in the dry form or ground in oil to form a paste.

MANUFACTURE

Process.

2. (*a*) *Dry Pigment.*—The pigment shall be the sublimed product prepared from lead sulfide ores, and shall be free from impurities and adulteration.

(*b*) *Paste.*—The paste shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) The color and color strength, when specified, shall be equal to that of a sample mutually agreed upon by buyer and seller.

(*b*) *Dry Pigment.*—The pigment shall conform to the following requirements:

	Maximum	Minimum
Coarse particles retained on a No. 325 sieve,[2] per cent	1.0	....
Lead oxide, per cent	18.0	11.0
Zinc oxide, per cent	9.0	....
Total impurities, including moisture, per cent	1.0	....

The remainder shall be lead sulfate.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

[2] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

(*c*) *Paste.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile mineral spirits or any combination of these substances.

The paste shall conform to the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	91.0	89.0
Linseed oil, per cent	11.0	9.0
Moisture and other volatile matter, per cent	0.7	....
Coarse particles and skins (total residue retained on a No. 325 sieve,[1] based on pigment), per cent	1.5	....

Number of Tests.

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

[1] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

STANDARD SPECIFICATIONS
FOR
RED LEAD[1]

Serial Designation: D 83 – 24

These specifications are issued under the fixed designation D 83; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1924.

Scope.

1. These specifications cover red lead to be used as a pigment, purchased in the form of dry pigment or ground in oil to form a paste.

MANUFACTURE

Process.

2. (*a*) *Dry Pigment.*—The pigment shall be made by roasting litharge or lead, or compounds of lead which yield litharge by heating.

(*b*) *Paste.*—The paste shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

NOTE.—Avoid storing red lead paste in places of high temperature, as heat accelerates the tendency of this material to cake or harden.

Purchasers are cautioned not to buy red lead in paste form unless it is to be used within three months after shipment by the contractor.

PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) *Dry Pigment.*—The pigment shall consist entirely of oxides of lead, free from all adulterants and shall conform to the following requirements:

	85-PER-CENT GRADE	95-PER-CENT GRADE
True red lead, Pb_3O_4, minimum, per cent	85.0	95.0
Total impurities, including moisture, soluble matter, water, and matter insoluble in a mixture of nitric acid and hydrogen peroxide, maximum, per cent	1.0	1.0
The remainder shall be lead monoxide (PbO)		
Coarse particles retained on a No. 325 sieve,[2] maximum, per cent	2.0	1.0

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

[2] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

When mixed with raw linseed oil, turpentine and liquid drier, in the proportions indicated below, the resulting paint, when brushed on a smooth vertical iron surface, shall dry hard and elastic without running, streaking or sagging:

Dry red lead	20 lb.
Raw linseed oil	5 pt.
Turpentine	2 gills
Liquid drier	2 "

(*b*) *Paste.*—The paste as shipped by the contractor, and for three months thereafter, shall not be caked in the container and shall readily break up in oil to form a smooth paint of brushing consistency.

The paste shall conform to the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	94.0	92.0
Linseed oil, per cent	8.0	6.0
Moisture and other volatile matter, per cent	0.5	...
Coarse particles and skins (total residue retained on a No. 325 sieve,[1] based on pigment), per cent	1.5	...

When mixed with raw linseed oil, turpentine and liquid drier in the proportions indicated below, the resulting paint, when brushed on a smooth vertical iron surface, shall dry hard and elastic without running, streaking or sagging:

Red lead paste	20 lb.
Raw linseed oil	4½ pt.
Turpentine	2 gills
Liquid drier	2 "

Number of Tests.

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

[1] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

STANDARD SPECIFICATIONS
FOR
MINERAL IRON OXIDE[1]

Serial Designation: D 84 – 27

These specifications are issued under the fixed designation D 84; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1927.

Scope.

1. These specifications cover iron oxide and iron hydroxide pigments of mineral origin and of red and brown colors.

MANUFACTURE

Process.

2. (*a*) *Dry Pigment.*—The pigment shall be very finely ground iron oxide or iron hydroxide or a mixture thereof. Siliceous minerals may be present and, if necessary, sufficient carbon pigment may be added to produce the desired color.

(*b*) *Paste.*—The paste shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) The mass color and character of the tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a sample mutually agreed upon by buyer and seller. The drying time with a vehicle which has been mutually agreed upon shall be equal to that of the agreed sample mentioned.

(*b*) *Dry Pigment.*—The pigment shall conform to the following requirements:

	Maximum	Minimum
Ferric oxide, per cent	...	30.0
Material other than ferric oxide, insoluble siliceous matter and loss on ignition, per cent	10.0	...
Coarse particles retained on a No. 325 sieve,[2] per cent	3.0	...

Organic coloring matters shall be absent.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

[2] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

(*c*) *Paste.*—The paste as received and three months thereafter shall not be caked in the container and shall break up readily in linseed oil to form a smooth paint of brushing consistency. It shall mix readily with linseed oil, turpentine or volatile mineral spirits, or any combination of these, without curdling.

The paste shall conform to the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	72.0	68.0
Linseed oil, per cent	32.0	28.0
Moisture and other volatile matter, per cent	0.7	....
Coarse particles and skins (total residue retained on a No. 325 sieve,[1] based on pigment), per cent	3.5	....

Number of Tests.

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

[1] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

STANDARD SPECIFICATIONS
FOR
OCHER[1]

Serial Designation: D 85 – 27

These specifications are issued under the fixed designation D 85; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1924; REVISED, 1927.

Scope.

1. These specifications cover ferrous earthy pigments, included under the general term "Ocher" and purchased either as dry pigment or ground in oil or in japan to form a paste.

MANUFACTURE

General Composition.

2. (*a*) *Dry Pigment.*—The pigment shall be a hydrated oxide of iron, permeating a siliceous base, and shall be free from added impurities and added coloring matter.

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

(*c*) *Paste in Japan.*—The paste in japan shall be made by thoroughly grinding the specified pigment in high-grade grinding japan.

PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) The mass color and character of the tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a sample mutually agreed upon by buyer and seller.

(*b*) *Dry Pigment.*—The pigment shall conform to the following requirements:

	Maximum	Minimum
Coarse particles retained on a No. 325 Sieve,[2] per cent.	1.0	..
Iron oxide (Fe_2O_3), per cent	...	17
Lime (CaO), per cent	5	..
Lead chromate, per cent	None	..
Organic colors, per cent	None	..

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

[2] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

(*c*) *Paste in Oil.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine or volatile mineral spirits, or any mixture of these substances.

The paste shall conform to the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	73	69
Linseed oil, per cent	31	27
Moisture and volatile matter, per cent	0.5	..
Coarse particles and skins (total residue retained on a No. 325 sieve,[1] based on pigment), per cent	1.5	..

(*d*) *Paste in Japan.*—The paste as received shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within one hour to a hard, flat coat that can be varnished within five hours of the time of application, without streaking or bleeding.

The paste shall conform to the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	73	69
Vehicle (japan), per cent	31	27
Coarse particles and skins (total residue retained on a No. 325 sieve,[1] based on pigment), per cent	1.5	..
Non-volatile matter in vehicle, per cent of the vehicle	....	40

Number of Tests.

4. One sample shall be taken at random from each lot of 1000 packages or fraction thereof. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

[1] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

STANDARD SPECIFICATIONS
FOR
LITHOPONE[1]

Serial Designation: D 208 – 26

These specifications are issued under the fixed designation D 208; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1925; ADOPTED IN AMENDED FORM, 1926.

Scope.

1. These specifications cover the pigment consisting of zinc sulfide and barium sulfate, commercially known as lithopone. The pigment may be purchased in the dry form or ground in oil to form a paste.

MANUFACTURE

Process.

2. (*a*) *Dry Pigment.*—The pigment shall be made by suitable treatment of a mixture of precipitated zinc sulfide and precipitated barium sulfate.

(*b*) *Paste.*—The paste shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) The behavior on exposure to light, the mixing properties with an approved vehicle, the final consistency with this vehicle and the color shall be equal to, and the brightness and tinting strength (or hiding power) shall be not less than, those of a sample, mutually agreed upon by buyer and seller.

(*b*) *Dry Pigment.*—The pigment shall conform to the following requirements:

	Maximum	Minimum
Coarse particles and skins (total residue retained on a No. 325 sieve[2]), per cent	1.0	....
Zinc sulfide, per cent	...	26.0
Zinc oxide, per cent	2.0	....
Material soluble in water, per cent	0.8	....
Barium sulfate	98 per cent of the remainder.	

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

[2] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

(*c*) *Paste.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions without curdling, with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances.

The paste shall conform to the following requirements:

	Maximum	Minimum
Pigment, per cent	80.0	76.0
Linseed oil, per cent	24.0	20.0
Coarse particles and skins (total residue retained on a No. 325 sieve,[1] based on pigment), per cent	1.5	....

Number of Tests.

4. (*a*) *Paste.*—One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

(*b*) *Dry Pigment.*—Dry pigment shall be sampled by taking a portion from every tenth barrel or from every one hundredth bag of the dry pigment.

[1] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

STANDARD SPECIFICATIONS
FOR
LAMPBLACK[1]

Serial Designation: D 209 – 26

These specifications are issued under the fixed designation D 209; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1925; ADOPTED IN AMENDED FORM, 1926.

Scope.

1. These specifications cover the pigment commonly known as lampblack. The pigment may be purchased in the dry form, or ground in oil or in japan to form a paste.

MANUFACTURE

Process.

2. (*a*) *Dry Pigment.*—The pigment shall be made by burning oils or tars in such a manner as to form a deposit of carbon or soot. The pigment shall be high grade in every respect, shall be free from oil, greasy matter and from admixture of any other substance.

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

(*c*) *Paste in Japan.*—The paste in japan shall be made by thoroughly grinding the specified pigment in high-grade grinding japan.

PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) The color and tone shall be equal to, and the tinting strength not less than that of a sample mutually agreed upon by buyer and seller.

(*b*) *Dry Pigment.*—The pigment shall conform to the following requirements:

Coarse particles retained on a No. 325 sieve,[2] maximum, per cent	1.0
Ash, maximum, per cent	1.0
Benzol extract (which must be colorless), maximum, per cent	0.5
Tone when diluted with zinc oxide	Clear-blue-gray

(*c*) *Paste in Oil.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions without curdling, with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

[2] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

The paste shall conform to the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	..	25
Linseed oil, per cent	75	..
Material volatile at 105° C	0.7	..
Coarse particles and skins left on a No. 325 sieve[1] (per cent of the dry pigment)	1.0	..

(*d*) *Paste in Japan.*—The paste as received shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within one hour to a hard, flat coat. It shall conform to the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	..	25
Vehicle, per cent	75	..
Coarse particles and skins retained on a No. 325 sieve[1] (per cent of the dry pigment)	1.0	..
Non-volatile matter in the vehicle, per cent of the vehicle	..	40

Number of Tests.

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

[1] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

STANDARD SPECIFICATIONS
FOR
BONE BLACK[1]

Serial Designation: D 210 - 26

These specifications are issued under the fixed designation D 210; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1925; ADOPTED IN AMENDED FORM, 1926.

Scope.

1. These specifications cover the pigment commonly known as bone black, ivory black or drop black. It may be purchased in the dry form, or ground in oil or in japan to form a paste.

MANUFACTURE

Process.

2. (*a*) *Dry Pigment.*—The pigment shall be made by the calcination of bones and shall be unmixed with any other substance.

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

(*c*) *Paste in Japan.*—The paste in japan shall be made by thoroughly grinding the pigment in high-grade grinding japan.

PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) The color and tone shall be equal to, and the tinting strength not less than, that of a sample mutually agreed upon by buyer and seller.

(*b*) *Dry Pigment.*—The pigment shall conform to the following requirements:

Coarse particles retained on a No. 325 sieve,[2] maximum, per cent	2.0
Ash, maximum, per cent of pigment dried at 105° C	88.0
Ash insoluble in acids, maximum, per cent	1.0
Benzol extract (which must be colorless), maximum, per cent	0.5
Tone when diluted with zinc oxide	Clear-blue-gray

(*c*) *Paste in Oil.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions without curdling, with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

[2] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

The paste shall conform to the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	..	45
Linseed oil, per cent	55	..
Coarse particles and skins (total residue retained on a No. 325 sieve,[1] based on pigment), per cent	2.5	..

(*d*) *Paste in Japan.*—The paste as received shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within one hour to a hard, flat coat. It shall conform to the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	..	45
Vehicle (japan), per cent	55	..
Coarse particles and skins (total residue retained on a No. 325 sieve,[1] based on pigment), per cent	2.5	..
Non-volatile matter in the vehicle, per cent of the vehicle	..	40

Number of Tests.

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

[1] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

STANDARD SPECIFICATIONS
FOR
CHROME YELLOW[1]

Serial Designation: D 211 – 27

These specifications are issued under the fixed designation D 211; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1925; ADOPTED IN AMENDED FORM, 1927.

Scope.

1. These specifications cover the pigments commonly known as lemon chrome yellow, medium chrome yellow, and orange chrome yellow. The pigment may be purchased in the dry form or ground in oil, or in japan, to form a paste.

MANUFACTURE

Process.

2. (*a*) *Dry Pigments.*—The dry pigments shall be chemical precipitates consisting of normal or basic lead chromates or mixtures of these with or without admixtures of other insoluble compounds of lead, but without any other admixtures.

(*b*) *Pastes in Oil.*—The pastes in oil shall be made by thoroughly grinding the specified pigments with pure raw or refined linseed oil.

(*c*) *Pastes in Japan.*—The pastes in japan shall be made by thoroughly grinding the specified pigments in high-grade grinding japan.

PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) The mass color and character of the tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a sample mutually agreed upon by buyer and seller.

(*b*) *Dry Pigments.*—The pigments shall conform to the following requirements:

Total matter soluble in water, maximum, per cent	0.5
Total of all substances other than insoluble compounds of lead, maximum, per cent	3.0
Organic colors or lakes	None
Coarse particles (total residue on a No. 325 sieve[2]), per cent, not over	1.0

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.TM. Committee D-1 on Preservative Coatings for Structural Materials.

[2] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

(*c*) *Pastes in Oil*—The pastes as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. They shall mix readily in all proportions without curdling, with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances.

The pastes shall conform to the following requirements:

Pigment, minimum, per cent	75
Linseed oil, maximum, per cent	25
Water and other volatile matter, maximum, per cent	0.7
Coarse particles and skins (total residue retained on a No. 325 sieve[1]), maximum, per cent of the pigment	1.5

(*d*) *Pastes in Japan.*—The pastes as received shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within one hour to a hard flat coat that can be varnished within five hours of the time of application without streaking or bleeding.

The pastes shall conform to the following requirements:

Pigment, minimum, per cent	70
Vehicle (japan), maximum, per cent	30
Coarse particles and skins (total residue retained on a No. 325 sieve[1]), maximum, per cent of the pigment	1.5
Non-volatile matter in the vehicle, minimum, per cent of the vehicle	40

Number of Tests.

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

[1] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

STANDARD SPECIFICATIONS
FOR
PURE CHROME GREEN[1]

Serial Designation: D 212 – 27

These specifications are issued under the fixed designation D 212; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1925; ADOPTED IN AMENDED FORM, 1927.

1. These specifications cover what is commercially known as pure chrome green. The pigment may be purchased in the dry form or ground in oil or in japan to form a paste. **Scope.**

MANUFACTURE

2. (*a*) *Dry Pigment.*—The pigment shall be a precipitated mixture of lead chromate and iron ferro and ferricyanide blue, with or without other insoluble compounds of lead. **Process.**

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

(*c*) *Paste in Japan.*—The paste in japan shall be made by thoroughly grinding the specified pigment with high-grade grinding japan.

PROPERTIES AND TESTS

3. (*a*) The mass color and character of the tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a sample mutually agreed upon by buyer and seller. **Composition and Properties.**

(*b*) *Dry Pigment.*—The pigment shall conform to the following requirements:

Percentage of total lead present in the form of chromate, minimum	70
Total impurities, maximum, per cent	3
Coarse particles (total residue retained on a No. 325 sieve[2]), maximum, per cent	1.0

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

[2] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

(*c*) *Paste in Oil.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances.

The paste shall conform to the following requirements:

Pigment, minimum, per cent	70
Linseed oil, maximum, per cent	30
Coarse particles and skins (total residue retained on a No. 325 sieve[1]), maximum, per cent of the pigment	1.5

(*d*) *Paste in Japan.*—The paste as received shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within one hour to a hard, flat coat that can be varnished within five hours of the time of application without streaking or bleeding.

The paste shall conform to the following requirements:

Pigment, minimum, per cent	65
Vehicle (japan), maximum, per cent	35
Coarse particles and skins (total residue retained on a No. 325 sieve[1]), maximum, per cent of the pigment	1.5
Non-volatile matter in vehicle, minimum, per cent of the vehicle	40

Number of Tests.

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

[1] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

STANDARD SPECIFICATIONS
FOR
REDUCED CHROME GREEN[1]

Serial Designation: D 213 – 27

These specifications are issued under the fixed designation D 213, the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1925; ADOPTED IN AMENDED FORM, 1927.

Scope.

1. These specifications cover what is known commercially as reduced chrome green, also known as grinders green. The pigment may be purchased in the dry form or ground in oil or in japan to form a paste.

MANUFACTURE

Process.

2. (*a*) *Dry Pigment.*—The pigment shall be a mixture of lead chromate and iron ferro and ferricyanide blue, with or without other insoluble compounds of lead, precipitated on a base of barium sulfate or insoluble siliceous material or any mixture thereof.

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

(*c*) *Paste in Japan.*—The paste in japan shall be made by thoroughly grinding the specified pigment in high-grade grinding japan.

PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) The mass color and character of the tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a sample mutually agreed upon by buyer and seller.

(*b*) *Dry Pigment.*—The pigment shall conform to the following requirements:

	MAXIMUM	MINIMUM
Sum of the barium sulfate and insoluble siliceous material, per cent	80	..
Color (total of insoluble lead compounds and iron blue), per cent	..	20
Percentage of total lead present in the form of chromate	..	70
Total calcium oxide in any form soluble in acid, per cent	1.0	..
Coarse particles (total residue retained on a No. 325 sieve[2]), per cent	1.5	..

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

[2] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

(*c*) *Paste in Oil.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions without curdling, with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances.

The paste shall conform to the following requirements:

Pigment, minimum, per cent	80
Linseed oil, maximum, per cent	20
Coarse particles and skins (total residue retained on a No. 325 sieve[1]), maximum, per cent of the pigment	2.0

(*d*) *Paste in Japan.*—The paste as received shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within one hour to a hard flat coat that can be varnished within five hours of the time of application without streaking or bleeding.

The paste shall conform to the following requirements:

Pigment, minimum, per cent	75
Vehicle (japan), maximum, per cent	25
Coarse particles and skins (total residue retained on a No. 325 sieve[1]), maximum, per cent	2.0
Non-volatile matter in vehicle, minimum, per cent of the vehicle	40

Number of Tests.

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

[1] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

STANDARD METHODS OF SAMPLING AND TESTING TURPENTINE[1]

Serial Designation: D 233 – 26

These methods are issued under the fixed designation D 233; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1926.

DETECTION AND REMOVAL OF SEPARATED WATER

1. Draw a portion by means of a glass or metal container with a removable stopper or top, or with a "thief," from the lowest part of the container, or by opening the bottom valve of the perfectly level tank car. If water is found to be present, draw it all out, record the quantity, and deduct it from the total volume of liquid delivered. Water Removal.

SAMPLING

2. The method of sampling given under (*a*) shall be used whenever feasible. When method (*a*) is not applicable, method (*b*), (*c*), or (*d*) shall be used according to the special conditions that obtain. Sampling.

(*a*) *While Loading Tank Car or While Filling Containers for Shipment.*—Samples shall be drawn by the purchaser's inspector at the discharge pipe where it enters the receiving vessel or vessels. The composite sample shall be not less than 5 gal. and shall consist of small portions of not more than 1 qt. each taken at regular intervals during the entire period of loading or filling.

The composite sample thus obtained shall be thoroughly mixed and from it three samples of not less than 1 qt. each shall be placed in clean, dry, glass bottles or tin cans, which shall be nearly filled with the sample and securely stoppered with new, clean corks or well-fitting covers or caps. These shall be sealed and distinctly labeled by the inspector; one shall be delivered to the purchaser, one to the seller, and the third held for check in case of dispute.

(*b*) *From Loaded Tank Car or Other Large Vessel.*—The composite sample taken shall be not less than 5 gal. and shall consist of numerous small samples of not more than 1 qt. each taken from the top, bottom, and intermediate points by means of a metal or glass container with removable stopper or top. This device, attached to a suitable pole,

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

is lowered to the various desired depths, when the stopper or top is removed and the container allowed to fill. The sample thus obtained is handled as in method (*a*).

(*c*) *Barrels and Drums.*—Barrels and drums shall be sampled after gaging the contents. Five per cent of the packages in any shipment or delivery shall be represented in the sample. Thoroughly mix the contents of each barrel to be sampled by stirring with a clean rod and withdraw a portion from about the center by means of a "thief" or other sampling device. The composite sample thus obtained shall be not less than 3 qt., shall consist of equal portions of not less than ½ pt. from each package sampled, and shall be handled as in method (*a*). Should the inspector suspect adulteration, he shall draw the samples from the suspected packages.

(*d*) *Small Containers, Cans, etc., of 10 gal. or Less.*—These shall be sampled, while filling, by method (*a*) whenever possible; but in case this is impossible the composite sample taken shall be not less than 3 qt. This shall be drawn from at least five packages (from all when fewer), and in no case from less than 2 per cent of the packages. The composite sample thus taken shall be thoroughly mixed and subdivided as in method (*a*).

LABORATORY EXAMINATION

Appearance. 3. Examine to determine compliance with the specifications.

Color. 4. Fill a 200-mm. perfectly flat-bottomed colorimeter tube, graduated in millimeters, to a depth of from 40 to 50 mm. with the turpentine to be examined. Place the tube in a colorimeter and place on it or under it a No. 2 yellow Lovibond glass. Over or under a second graduated tube in the colorimeter, place a No. 1 yellow Lovibond glass and run in the same turpentine until the color matches as nearly as possible the color in the first tube. Read the difference in depth of the turpentine in the two tubes. If this difference is 50 mm. or more, the turpentine is "Standard" or better.

Odor. 5. Determine odor by comparison with the agreed-upon sample which shall have been kept in the dark in completely filled well-stoppered bottles and free from separated water. In case no agreed-upon sample is available, compare with samples of known purity that have been kept in filled stoppered bottles in the dark and are free from separated water.

Specific Gravity. 6. Determine specific gravity at 15.5/15.5° C., by any convenient method that is accurate within 2 points in the fourth figure.

Refractive Index. 7. Determine refractive index at 20° C. with an accurate instrument. When the refractive index is determined at any other tem-

perature, the readings obtained shall be corrected to 20° C. by adding to or by subtracting from the actual reading 0.00045 for each degree Centigrade that the temperature at which the determination was made is, respectively, above or below 20° C.

8.—*Apparatus.*[1]—(*a*) *Condenser.*—The type of apparatus is shown in Fig. 1, or may be the apparatus described in the Standard Distillation.

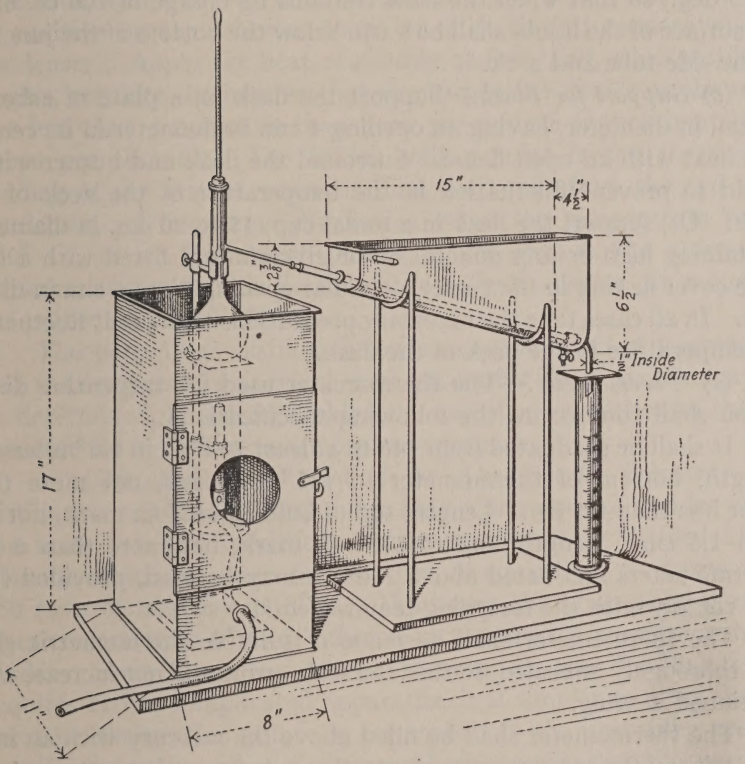

FIG. 1.—Distillation Apparatus.

Method of Test for Distillation of Gasoline, Naphtha, Kerosine and Similar Petroleum Products (Serial Designation: D 86) of the American Society for Testing Materials,[2] substituting for the thermometer there described an immersed thermometer, such as is described below.

[1] The flask, condenser, shield, ring support and hard asbestos board, gas burner or electric heater, described in the Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine and Similar Petroleum Products (Serial Designation: D 86) of the American Society for Testing Materials, see p. 378, which are essentially the same as the equipment herewith described, may be employed in the distillation test wherever it is available.

[2] See p. 378.

(*b*) *Flask.*—Comparable results can be obtained only by using flasks of the same dimensions. The distilling flask used shall be the standard Engler flask, as used for petroleum distillation, having the following dimensions: Diameter of bulb, 6.5, cm.; cylindrical neck, 15 cm. long, 1.6 cm. internal diameter; side or vapor tube, 10 cm. long, 0.6 cm. external diameter, attached to neck at an angle of 75 deg., so that when the flask contains its charge of 100 cc. of oil the surface of the liquid shall be 9 cm. below the bottom of the junction of the side tube and neck.

(*c*) *Support for Flask.*—Support the flask on a plate of asbestos 20 cm. in diameter, having an opening 4 cm. in diameter in its center, and heat with an open flame. Surround the flask and burner with a shield to prevent fluctuation in the temperature of the neck of the flask. Or, support the flask in a metal cup, 15 to 20 cm. in diameter, containing high-boiling mineral oil or glycerin and fitted with a concave cover having in the center a circular opening 5½ to 6 cm. in diameter. In all cases take the necessary precautions to prevent fluctuation in temperature in the neck of the flask.

(*d*) *Thermometer.*—The thermometer used for turpentine distillation shall conform to the following specifications:

It shall be graduated from 145 to at least 200° C. in 0.2° intervals. Length, bottom of thermometer to 175° C. mark, not more than 8 nor less than 6.5 cm. Length, top of bulb to 145° C. mark, not less than 1.5 cm. Length, 145 to 175° C. mark, not more than 6 cm. Thermometers graduated above 200° C. may be used, provided they also comply with the foregoing requirements.

The thermometer shall be made of suitable thermometric glass and thoroughly annealed, so that the scale errors will not increase after continued heating.

The thermometer shall be filled above the mercury with an inert gas, with sufficient pressure above the mercury column to prevent breaking of the column. It shall have a reservoir at the top, so that the pressure will not become excessive at the highest temperature.

Every fifth graduation shall be longer than the intermediate ones, and the marks shall be numbered at each interval of 5° C. The graduation marks shall be clear-cut and fine and the numbering clear-cut and distinct.

The error at any point on the scale shall not exceed ±0.5° C. when tested for total immersion of the mercury column.

(*e*) *Receiving Cylinder.*—Collect the distillate in an accurately graduated 50 or 100-cc. cylinder. The so-called normal or precision cylinder of 50 cc. capacity, having an internal diameter of 1.5 cm. and

graduated in 0.2 cc., is preferred. If a cylinder with larger inside diameter is used, a pasteboard cover should be placed over the top and surround the condenser tube.

Method.

9. *Operation.*—Place 100 cc. of the turpentine and several small pieces of pumice (or glass) in the distilling flask, fit the thermometer so that the top of the mercury bulb is level with the bottom of the side tube, and the 175° C. mark is below the cork. Place the flask in position on the asbestos board or oil bath and connect with the condenser. Apply the heat cautiously at first, and, when distillation begins, regulate the heat so that the turpentine distills at the rate of not less than 4 nor more than 5 cc. per minute (approximately two drops per second). The initial boiling point is the thermometer reading at the instant when the first drop falls from the end of the condenser. Discontinue distillation when the temperature reaches 170° C., or an equivalent thereof, depending on the atmospheric pressure, as described in Section 10; let the condenser drain and read the percentage distilled.

The percentage distilled below successive selected temperatures and the temperature at which each successive 10 cc. distills may also be determined, if desired, making the necessary correction of the temperature for variations in atmospheric pressure.

Barometer Reading Correction.

10. *Correction for Variation in Atmospheric Pressure.*—Since distillation results are comparable only when obtained under exactly the same pressure conditions, turpentine shall be distilled at that pressure which, at room temperature, is equivalent to a pressure of 760 mm. of mercury at 0° C. Whenever the atmospheric pressure after correcting to 0° C. is other than 760 mm., a correction shall be made. Since alteration of the pressure in the distilling system requires rather complicated apparatus, it is simpler to alter the temperature observation points to correspond to the prevailing pressure.

To determine what the atmospheric pressure at the prevailing room temperature, or at the temperature of the barometer, would be at 0° C., read the barometer and the thermometer alongside when about to begin distillation. Referring to Table I, under the column nearest the observed pressure reading and on the line nearest the observed temperature of the barometer will be found the correction which must be subtracted from the observed pressure reading to obtain the equivalent, or true, reading at 0° C.

The distilling temperature of turpentine is affected plus (+) or minus (−) 0.057° C. for each millimeter variation of the barometer above or below the normal 760 mm. at 0° C.[1] If the barometer reading,

[1] Landolt-Börnstein Physikalisch-Chemische Tabellen, Ed. 4, Table 127, p. 435.

after correcting to 0° C., is below 760 mm., the turpentine will distill at a slightly lower temperature than under normal pressure. Therefore, the temperature recorded at the beginning of distillation (and any others observed during the course of the distillation) must be corrected to get its equivalent at normal pressure. The final temperature observation point (170° C., Section 9) must be altered accordingly to get its equivalent at the pressure (corrected to 0° C.) at which distillation is made.

For example, if the barometer reading, after correcting to 0° C., is 750 mm., the correction of the observed initial distilling temperature

TABLE I.—CORRECTION TO BAROMETER READINGS.[1]

(From Circular F, Instrument Division, Weather Bureau, U. S. Department of Agriculture.)

Temperature, deg. Cent.	Observed Reading of Barometer, mm.														
	640	650	660	670	680	690	700	710	720	730	740	750	760	770	780
15.0	1.56	1.59	1.61	1.64	1.66	1.69	1.71	1.74	1.76	1.78	1.81	1.83	1.86	1.88	1.91
16.0	1.67	1.69	1.72	1.75	1.77	1.80	1.83	1.85	1.88	1.90	1.93	1.96	1.98	2.01	2.03
17.0	1.77	1.80	1.83	1.86	1.88	1.91	1.94	1.97	1.99	2.02	2.05	2.08	2.10	2.13	2.16
18.0	1.88	1.91	1.93	1.96	1.99	2.02	2.05	2.08	2.11	2.14	2.17	2.20	2.23	2.26	2.29
19.0	1.98	2.01	2.04	2.07	2.10	2.13	2.17	2.20	2.23	2.26	2.29	2.32	2.35	2.38	2.41
20.0	2.08	2.12	2.15	2.18	2.21	2.25	2.28	2.31	2.34	2.38	2.41	2.44	2.47	2.51	2.54
21.0	2.19	2.22	2.26	2.29	2.32	2.36	2.39	2.43	2.46	2.50	2.53	2.56	2.60	2.63	2.67
22.0	2.29	2.33	2.36	2.40	2.43	2.47	2.51	2.54	2.58	2.61	2.65	2.69	2.72	2.76	2.79
23.0	2.40	2.43	2.47	2.51	2.54	2.58	2.62	2.66	2.69	2.73	2.77	2.81	2.84	2.88	2.92
24.0	2.50	2.54	2.58	2.62	2.66	2.69	2.73	2.77	2.81	2.85	2.89	2.93	2.97	3.01	3.05
25.0	2.60	2.64	2.68	2.72	2.77	2.81	2.85	2.89	2.93	2.97	3.01	3.05	3.09	3.13	3.17
26.0	2.71	2.75	2.79	2.83	2.88	2.92	2.96	3.00	3.04	3.09	3.13	3.17	3.21	3.26	3.30
27.0	2.81	2.85	2.90	2.94	2.99	3.03	3.07	3.12	3.16	3.20	3.25	3.29	3.34	3.38	3.42
28.0	2.91	2.96	3.00	3.05	3.10	3.14	3.19	3.23	3.28	3.32	3.37	3.41	3.46	3.51	3.55
29.0	3.02	3.06	3.11	3.16	3.21	3.25	3.30	3.35	3.39	3.44	3.49	3.54	3.58	3.63	3.68
30.0	3.12	3.17	3.22	3.27	3.32	3.36	3.41	3.46	3.51	3.56	3.61	3.66	3.71	3.75	3.80
31.0	3.22	3.27	3.32	3.37	3.43	3.48	3.53	3.58	3.63	3.68	3.73	3.78	3.83	3.88	3.93
32.0	3.33	3.38	3.43	3.48	3.54	3.59	3.64	3.69	3.74	3.79	3.85	3.90	3.95	4.00	4.05
33.0	3.43	3.48	3.54	3.59	3.64	3.70	3.75	3.81	3.86	3.91	3.97	4.02	4.07	4.13	4.18

[1] These corrections apply to a mercurial barometer with brass scale. They can, however, be used for a mercurial barometer with glass scale, since the errors introduced thereby are negligible as applied to the work contemplated in this circular For exact correction to be applied to such a barometer see Smithsonian Physical Tables, p. 119 (1914). An aneroid barometer should not be relied on.

For barometer readings below 640 mm. the correction can be interpolated, since the difference at any particular temperature for each 10 mm. variation in barometer reading is practically constant.

will be $0.057 \times 10 = 0.6°$ C. approximately. If the reading of the thermometer when the turpentine begins to distill is 155.6° C., the corrected initial distilling temperature will be $155.6° + 0.6° = 156.2°$ C. Furthermore, the temperature observation point at end of distillation (170° C. at 760 mm.) must be altered to the same extent. Since the turpentine is distilling 0.6° C. below what it would at normal pressure, distillation must be discontinued at 0.6° C. below the specified limit of 170° C. to determine the percentage distilling below 170° C.

If the barometer reading corrected to 0° C. is above 760 mm., subtract the temperature correction from the observed thermometer reading to determine the initial distilling point, and continue distilla-

tion to 170° C. plus the correction to determine the percentage distilling below 170° C.

11. (*a*) Place 20 cc. of 38 *N* (equivalent to 100.92 per cent H_2SO_4) sulfuric acid in a graduated, narrow-necked Babcock flask, stopper, and place in ice water to cool. Add slowly, from a pipette, 5 cc. of the turpentine to be examined. Gradulaly mix the contents, keeping warm, but being very careful that the temperature does not rise above 60° C. When the mixture no longer warms up on shaking, agitate thoroughly and place the flask in a water bath and heat at 60 to 65° C. for not less than 10 minutes, keeping the contents of the flask thoroughly mixed by vigorous shaking for one-half minute each time, six times during the period. Do not stopper the flask after the turpentine has been added, as it may explode. Cool to room temperature, fill the flask with concentrated sulfuric acid until the unpolymerized oil rises into the graduated neck and centrifuge from four to five minutes at not less than 1200 r. p. m., or for 15 minutes at 900 r. p. m., or allow to stand, lightly stoppered, for 12 hours. Calculate the percentage, note the consistency and color, and determine the refractive index (at 20° C.) of the unpolymerized residue. Polymerization.

(*b*) *Reagent for Testing.*—In a weighed glass-stoppered bottle (the regular 2½-liter acid bottle is of a convenient size) mix ordinary concentrated sulfuric acid (sp. gr. 1.84) with fuming sulfuric acid. If the fuming acid used contains 50 per cent excess SO_3, the ratio of one part, by weight, of the former to three-fourths of a part, by weight, of the latter will give a mixture slightly stronger than the required strength. To determine the exact strength of this mixture in terms of H_2SO_4, weigh exactly, in a weighing pipette of about 10 cc. capacity, approximately 20 g. of the acid. Allow it to flow down the sides of the neck into a 1000-cc. volumetric flask containing about 200 cc. of distilled water. When the pipette has drained, wash all traces of the acid remaining in the pipette into the flask, taking precautions to prevent loss of SO_3, and make up to the mark. Titrate 20-cc. portions, drawn from a burette, against half normal alkali. Calculate the concentration in terms of the percentage of H_2SO_4 in the sample taken.

In the same way determine the percentage of H_2SO_4 in the stock of ordinary concentrated acid (sp. gr. 1.84). From these data calculate the quantity of the latter which must be added to the quantity of mixed acid in the weighed bottle to bring it to a concentration, in terms of H_2SO_4, of 100.92 per cent.

After adjusting the concentration by the addition of the ordinary sulfuric acid, thoroughly shake the bottle of mixed acid and again determine its concentration. The allowable variation is ± 0.05 per

cent H_2SO_4. Finally as a check run a polymerization test on gum turpentine known to be pure. The residue should fall below 2 per cent.

Special precautions must be taken to prevent dilution of this acid by the absorption of atmospheric moisture. The arrangement shown in Fig. 2 is most suitable for storing and delivering measured quantities of this reagent.

With the three-way stopcocks *A* and *B* in the position shown, acid is siphoned into the pipette *P*, the displaced air passing into *R*. To

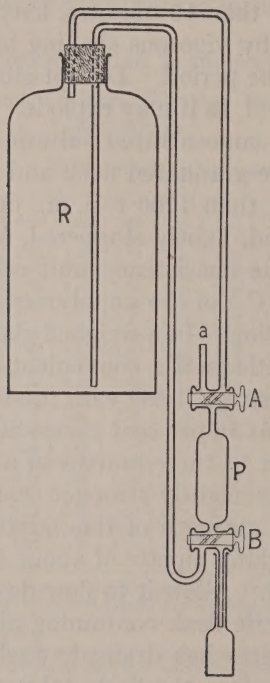

FIG. 2.—Acid Bottle and Pipette.

empty the pipette, *A* and *B* are turned to the position shown by the broken lines, air passing in at *a*. The acid adhering to the walls of the pipette dries this air so that when it passes into *R* on again filling the pipette there is no accumulation of moisture in the acid remaining in the reservoir. If such arrangement is not to be had, the acid should be kept in well-fitting glass-stoppered bottles of not more than one-half liter capacity.

STANDARD METHODS OF TEST
FOR
SPECIFIC GRAVITY OF PIGMENTS[1]

Serial Designation: D 153 – 27

These methods are issued under the fixed designation D 153; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED, 1924; REVISED, 1927.

METHOD *A:* FOR ROUTINE TESTING OF SEVERAL SAMPLES SIMULTANEOUSLY

APPARATUS

1. The apparatus shall consist of the following:

(*a*) A pyknometer as shown in Fig. 1 (*a*) or (*b*) having a capacity of 50 cc. **Apparatus.**

(*b*) A water bath consisting of a vessel filled with sufficient water to permit of only a very gradual rise in temperature and equipped with a stirring device, preferably air-blown.

(*c*) An open-tube manometer, see *f*, Fig. 3, made of glass tubing 6 mm. in diameter, filled with mercury to approximately 86 cm., fitted with rubber pressure-tubing attached to a T-joint leading to the desiccator and the pump.

NOTE.—The difference in levels of the mercury in the manometer when the system is in operation, subtracted from the barometer reading taken at the same time, gives the vacuum of the system in millimeters of mercury. The difference between the barometer and the manometer reading should not exceed 3 mm.

(*d*) A glass desiccator, having a hole at the side, constructed with heavy walls to withstand a vacuum of one atmosphere.

(*e*) A laboratory water vacuum pump to expel the greater portion of air in the desiccator.

(*f*) An oil vacuum pump with motor to give a vacuum of not more than 3 mm.

NOTE.—The "Hyvac" oil pump is satisfactory. This pump has a displacement of approximately 200 cu. in. per minute or 7 cu. ft. per hour at a speed of 900 r.p.m. It will evacuate a flask of one-liter capacity to a vacuum of 1 mm. in two minutes.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

(*g*) A weighing bottle with cork. The neck shall be small enough to fit inside the neck of the pyknometer. This latter requirement is essential, since small quantities of pigment easily adhere to the ground-glass joint of the pyknometer.

(*j*) A thermometer having a range of from 0 to 60° C. graduated in 0.1° C.

PROCEDURE

Standardization of Pyknometers.

2. The pyknometer shall be filled with freshly-boiled distilled water and brought to a temperature of 15.6° C. It shall be dried and weighed as specified in Section 7. It shall then be cleaned, weighed, and dried. It shall then be filled with the kerosine to be used in

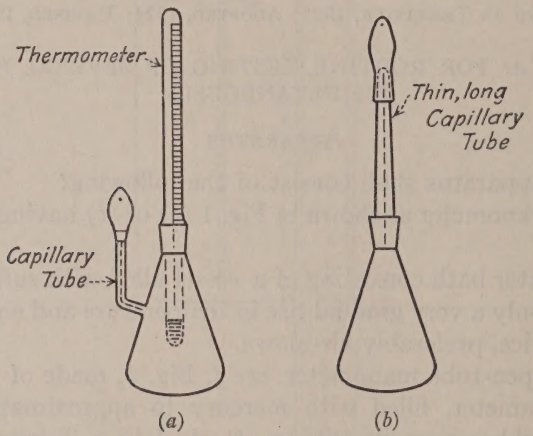

FIG. 1.—Pyknometer.

making the test and again brought to a temperature of 15.6° C., and dried and weighed in the same manner as before. The specific gravity of the kerosine shall be calculated from the formula:

$$\text{Specific Gravity } 15.5°/15.5° \text{ C.} = \frac{\text{Weight of Kerosine}}{\text{Weight of Water}}$$

Drying.

3. The pigment shall be dried in an oven at 105° C. for 2 hours.

NOTE.—It is preferable to use an electric oven for this purpose.

Weighing.

4. A sample of the pigment shall be weighed, by difference, in the weighing bottle. For blacks, blues, and lakes of light specific gravity, about 1 g. should be used; for inert crystallin pigments, about 4 g.; for opaque white pigments, 7 to 10 g.; and for red lead, from 15 to 20 g. should be used.

NOTE.—Due to the hygroscopic nature of some of the pigments it is necessary to use a weighing bottle fitted with a cork stopper.

Transferring to Pyknometer.

5. Sufficient kerosine shall be poured into the pyknometer to form a $\frac{1}{4}$-in. layer in the bottom and a quantity of pigment from the weighing bottle shall be added reaching approximately three-fourths of the distance to the kerosine level. The kerosine shall always cover the pigment. The sample shall be stirred with a polished round-bottom glass rod until completely covered by the kerosine, more kerosine being added from the wash bottle if necessary. The rod shall be washed with kerosine.

Removal of Occluded Air.

6. The pyknometers shall be placed in the desiccator, which shall then be closed and attached to the water pump until the greater part of the air is expelled from the system. This takes from 5 to 10 minutes. The system shall then be closed with a pinch-cock and the desiccator attached to the oil pump for the removal of the small amounts of air given off at the low pressures obtainable with the oil pump. The manometer is used to indicate whether the oil pump is giving the proper vacuum. When the manometer indicates that the vacuum, which should not be greater than 3 mm., is constant, the oil pump may be cut off for short periods, precaution being taken that the vacuum does not change materially due to leakage. It will be noticed that bubbles of air come from the pigments very rapidly at first and that this action gradually decreases and finally stops altogether. The time required for complete removal of air varies from 30 minutes to two hours, depending upon the nature of the pigment. When no more bubbles can be seen, it is assumed that all the occluded air has been given off and that the pigment is thoroughly wet with kerosine. Air can then be slowly admitted to the desiccator by means of the pinch-cock.

Filling and Bringing to Temperature.

7. The pyknometer shall be taken from the desiccator and filled with kerosine, care being taken to add a sufficient quantity to prevent the formation of air bubbles when placement of the thermometer is made. The thermometer shall be placed in the water bath, which shall be cooled with ice to a temperature of between 10 and 13° C. The pyknometer shall be placed in the bath and be permitted to come to constant temperature. The pyknometer thermometer or capillary tube shall then be inserted. Enough warm water shall be added to the bath to raise the temperature suddenly to about 14.5° C. in order to expand the kerosine and prevent it from creeping down the capillary and admitting a small amount of air. The bath shall be allowed to come to a temperature of 15.6° C. The capillary tube shall be wiped with filter paper and the cap put on. All temperatures on the thermometer in the bath shall be read, but not on the thermometer in the

pyknometer. The pyknometer shall be removed from the bath and dried. It shall then be allowed to stand for 30 minutes to enable it to come to room temperature, and weighed.

NOTE.—It is advisable to allow the pyknometer to stand approximately the same time before each weighing so as to compensate for slight errors due to evaporation at the joints.

Specific Gravity.

8. The specific gravity shall be calculated from the formula:

$$\text{Specific Gravity of Pigment} = \frac{P \times S}{(P + K) - F}$$

where K = Weight of the bottle filled with kerosine only;
P = Weight of pigment used;
F = Final weight of the bottle with pigment and kerosine;
S = Specific gravity of the kerosine used.

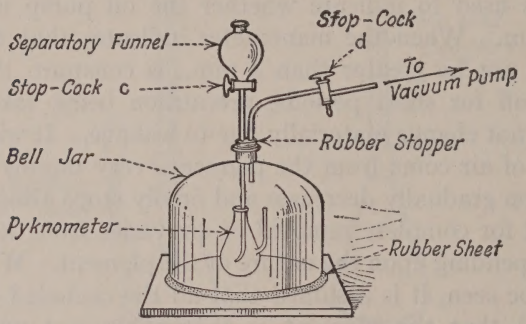

FIG. 2.

Precautions.

9. Before a new desiccator is used for the first time, it shall be wrapped in a towel and tested under the vacuum to be used, great care being exercised in handling the desiccator when the vacuum is on, as any sudden jar may cause it to collapse.

Number of Samples.

10. It has been found convenient to run six samples at one time, the desiccator specified being of the proper size to accommodate this number.

METHOD *B:* FOR TESTS REQUIRING HIGHEST ACCURACY

APPARATUS

Apparatus.

11. The apparatus shall consist of the following:

(*a*) A pyknometer as shown in Fig. 1 (*a*) or (*b*) having a capacity of 50 cc.

(*b*) A water bath consisting of a vessel filled with sufficient water to permit of only a very gradual rise in temperature and equipped with a stirring device, preferably air-blown.

(*c*) A glass bell jar with a two-hole rubber stopper. Into one hole of the stopper is fitted a separatory funnel with a well-ground stop-cock *c*, Fig. 2, and with the lower tube extending into the pyknometer (just below the opening of the side arm in Fig. 1 (*a*)). Into the other hole of the stopper is fitted a glass tube with a well-ground 3-way stop-cock *d*, Fig. 2, the tube connecting with the vacuum pump *e*, Fig. 3. The bell jar rests on a sheet of rubber, cemented or vulcanized to a glass or iron plate. With stop-cock *c* closed and stop-cock *d* opened to the pump, the system shall withstand and maintain a vacuum.

NOTE.—A suitable desiccator may be substituted for the bell jar.

(*d*) A thermometer having a range of from 0 to 60° C. graduated in 0.1° C.

(*e*) A high vacuum pump *e*, Fig. 3.

(*f*) An open-tube manometer *f*, Fig. 3, as described in Section 1 (*c*).

(*g*) A storage bottle *h*, Fig. 3, for kerosine or other wetting liquid.

PROCEDURE

12. The pyknometer containing the weighed sample of dried pigment shall be placed under the bell jar. Stop-cocks *c* and *d* shall be closed, the vacuum pump started and then stop-cock *d* gradually opened to the pump. When the air has all been removed, with a vacuum of 1 mm. maintained, the separatory funnel shall be filled with kerosine, stop-cock *d* closed, and at once stop-cock *c* shall be gradually opened, adding sufficient kerosine to completely cover the pigment. The pump shall be stopped and suction at *d* released. Finally, the pyknometer shall be filled with kerosine and the test completed as described in Sections 7 and 8 under Method *A*. Procedure.

NOTES

1. Before a new bell jar (or desiccator) is used for the first time, it shall be tested under a vacuum as described in Section 9.

2. Stop-cock *c* must be well-ground and should be lubricated with castor oil or glycerin. See precautions in Section 19 under Method *C* concerning the necessity of testing the system for leaks before making a determination.

3. With certain pigments that are not wetted well with kerosine, the substitution of turpentine has been found very efficient. However, when turpentine, or any other liquid having a high evaporation rate, is used, a pyknometer of type *a* or *b*, Fig. 1, is not satisfactory, on account of losses around the ground-glass joints. When using such liquids as turpentine, the bottle (as a pyknometer) and apparatus as described in Section 13 under Method *C* should be used, except that instead of attempting to measure an accurate volume of the liquid from the burette, the bottle (plus the sample plus the wetting liquid) is finally weighed as in Method *B*.

METHOD *C*: RAPID AND ACCURATE TESTING OF SINGLE SAMPLES

APPARATUS

Apparatus.

13. The apparatus shall consist of the following (see Fig. 3):

(*a*) A 100-cc. burette with a 75-cc. bulb in the upper part and the lower part (25 cc.) graduated in 0.05 cc. (see Fig. 4).

(*b*) A special 100-cc. graduated flask, *b*, Fig. 3, with ground-glass stopper. The flask shall be thick enough to withstand and maintain a vacuum and should weigh less than 35 g. The neck of the flask

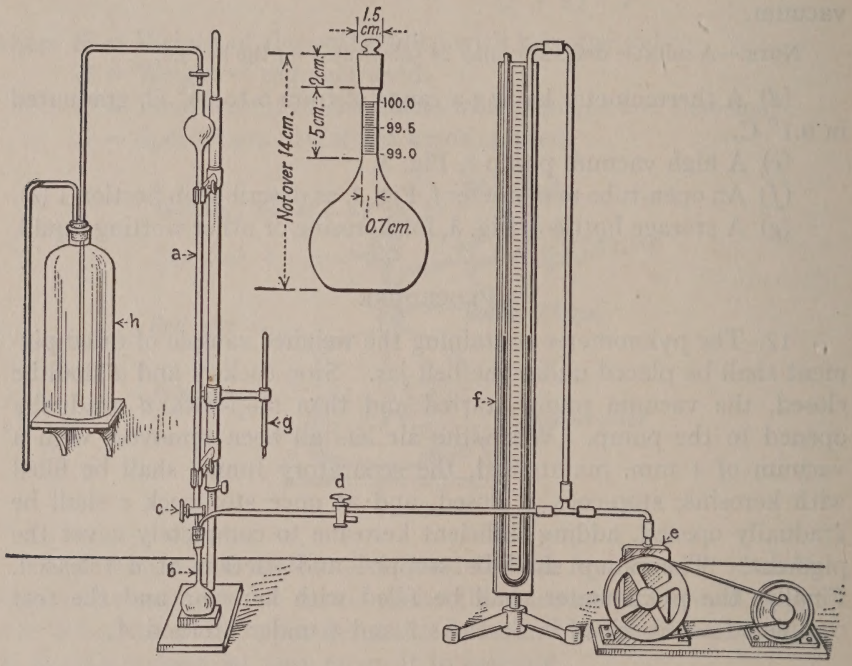

FIG. 3.—Apparatus Assembly for Determining Specific Gravity of Pigments.

should be graduated in 0.05 cc. between the 99 and 100-cc. marks. The dimensions of the flask are shown in Fig. 3.

(*c*) A tightly-ground stop-cock *c*, in the burette *a*.

(*d*) A 3-way stop-cock *d*, connecting with the vacuum pump *e*.

(*e*) A high vacuum pump *e*, of the type requiring no preliminary or "backing" pump.

NOTE.—The "Hyvac" oil pump is satisfactory.

(*f*) An open-tube manometer *f*, Fig. 3, as described in Section 1 (*c*).

NOTE.—The difference in levels of the mercury in the manometer when the system is in operation, subtracted from the barometer reading taken at the same

time, gives the vacuum of the system in millimeters of mercury. The difference between the barometer and the manometer readings should not exceed 1 mm.

(*g*) A thermometer *g*, having a range of from 0 to 60° C. graduated in 0.1° C.

(*h*) A storage bottle *h*, for kerosine or other wetting liquid.

PROCEDURE

14. The flask shall be connected to the burette and pump by means of a two-hole rubber stopper. The system shall be evacuated

Standardization of Apparatus.

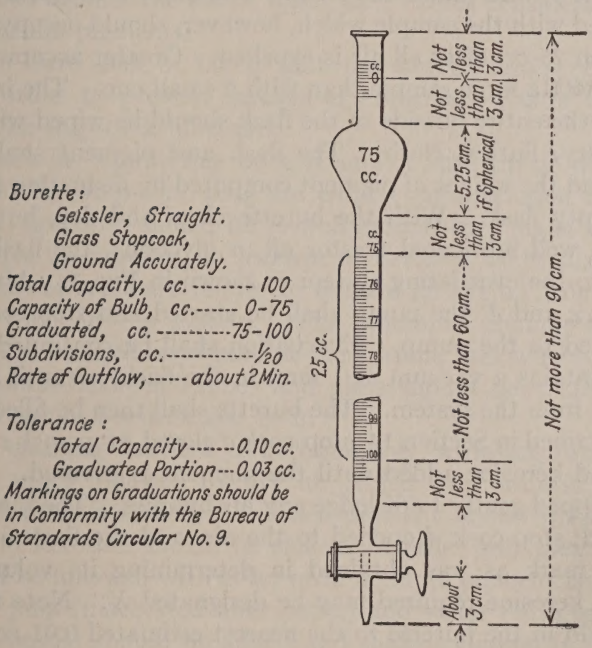
Burette:
Geissler, Straight.
Glass Stopcock,
Ground Accurately.
Total Capacity, cc. -------- 100
Capacity of Bulb, cc. ------ 0-75
Graduated, cc. ----------75-100
Subdivisions, cc. ----------1/20
Rate of Outflow, ------ about 2 Min.
Tolerance:
Total Capacity -------0.10 cc.
Graduated Portion---0.03 cc.
Markings on Graduations should be in Conformity with the Bureau of Standards Circular No. 9.
75 cc.
25 cc.
Not less than 3 cm.
Not less than 3 cm.
5.25 cm.
if Spherical
Not less than 3 cm.
Not less than 60 cm.
Not less than 3 cm.
About 3 cm.
Not more than 90 cm.

FIG. 4.—100-cc. Glass Burette.

with stop-cock *c* closed until the pump maintains a vacuum of 1 mm. in the flask (this requires only a few minutes). The 3-way stop-cock *d* shall be closed for 30 seconds, and again opened to the pump. There should be no appreciable change in the mercury levels in the manometer, indicating that the system beyond stop-cock *d* is tight. With the vacuum still maintained, the burette shall be filled from the top with kerosine, the level adjusted to the zero mark with a piece of capillary tubing. Stop-cock *d* shall now be closed and then stop-cock *c* carefully opened, admitting about 75 cc. of kerosine into the flask. Stop-cock *d* shall be opened to the air, thus releasing the

vacuum in the flask, and the flask shall be filled with kerosine to a definite mark on the neck. Read the burette, calling this reading K (the volume of the flask).

Procedure.

15. The flask shall then be cleaned with ether, dried, and weighed. A glass counterpoise having the same superficial area and treated the same way may be placed on the opposite pan in the balance. A quantity of the dry pigment to be tested shall be transferred to the flask by means of a clean, dry, glass funnel whose stem reaches to the bottom of the bulb. A piece of stiff nickel wire is convenient in pushing the powder down the stem. The bulb of the flask should be nearly filled with the sample which, however, should occupy a volume of less than 25 cc. after all air is expelled. Greater accuracy may be obtained with a large sample than with a small one. The inside stem as well as the entire outside of the flask should be wiped with a clean piece of dry, lintless cloth. The flask and pigment shall then be weighed and the weight of pigment computed by deducting the weight of the empty flask. With the burette clean and dry, but with the stop-cocks well lubricated (castor oil or glycerin), the flask shall be attached to the evacuating system as shown in Fig. 3. After closing stop-cocks *c* and *d*, the pump shall be started and stop-cock *d* carefully opened to the pump. Evacuation shall be continued until the pump maintains a vacuum of 1 mm. in the flask, or until all the air is expelled from the system. The burette shall then be filled from the top as described in Section 14, stop-cock *d* closed, stop-cock *c* gradually opened and kerosine added until the pigment is covered. The flask shall be tapped gently to dislodge any air bubbles. The pump should be stopped, stop-cock *d* opened to the air, and the flask filled up to the same mark as was obtained in determining its volume. The volume of kerosine required may be designated X. Note the height of the liquid in the burette to the nearest estimated 0.01 cc.

NOTE.—The removal of all air can not be stressed too greatly, as the presence of air will cause low results.

Calculation.

16. The specific gravity shall be calculated from the formula:

$$\text{Specific Gravity of Pigment} = \frac{S}{K - X}$$

where S = weight of pigment used;
K = volume of kerosine required to fill the flask when empty;
X = volume of kerosine required to fill the flask when the pigment is present.

Temperature.

17. Since the specific gravity of a pigment is only slightly affected by temperature, such variations as occur under normal conditions in a

room would not materially affect the results. Care shall be taken, however, that the temperature of the liquid after transferring to the flask is approximately the same as it was when in the burette.

18. While kerosine has been found to be a good wetting medium, any liquid which does not have a high evaporation rate may be used. The liquids are interchangeable, as no constants on them need be determined. Hence, a pigment containing a dye which is slightly soluble in kerosine could be run with another liquid in the same apparatus without special standardization for that liquid. Operators are cautioned against the use of water as it causes considerable frothing with certain pigments. **Wetting Medium.**

19. (*a*) Care shall be taken that the burette stop-cock is well ground in order to prevent leakage of kerosine. Castor oil is suggested as the stop-cock lubricant. **Precautions.**

(*b*) Since in determining both *K* and *X*, the tip of the burette and bore of the stop-cock plug are empty, no correction is needed; stop-cock *c*, however, should be so well ground that under a vacuum of 1 mm. for 30 minutes no leakage of kerosine takes place. The usual sources of error are failure to remove all the air from the pigment and leaks in the system. The minimum amount of rubber tubing should be used anywhere in the system, and wherever this is used the joints between rubber and glass should be coated with a melted mixture of beeswax and rosin.

(*c*) In cleaning the flask of kerosine only, a rinsing two or three times with ether followed by dry air (dried over sulfuric acid and calcium chloride) is sufficient. When pigment is also present, both pigment and kerosine should be shaken together and then emptied. This should be followed with ether until no more pigment is removed. Some filter pulp (macerated filter paper) and water (with or without some glass beads) should be added and shaken vigorously. Repetition may be necessary. The flask should then be rinsed with distilled water and either dried in an oven or rinsed with alcohol and ether followed by dry air. In determining specific gravity by this method, there is no reason why the flask if made of Pyrex glass can not be heated, followed by cooling during evacuation if such heating has no effect on the sample.

STANDARD METHOD OF TEST
FOR
COARSE PARTICLES IN PAINT PIGMENTS[1]

Serial Designation: D 185 – 26

This method is issued under the fixed designation D 185; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1924; ADOPTED IN AMENDED FORM, 1926.

APPARATUS

Apparatus.

1. The apparatus shall consist of a 3-in. No. 325 (44-micron) sieve conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials.[2] A 3-in. No. 325 (44-micron) sieve for comparison purposes should be retained in the laboratory as a standard. Whenever a new sieve is secured, a practical test of its accuracy should be made by running on it and on the standard sieve a pigment that has a considerable percentage of coarse particles. A reserve stock of such pigment should be kept for this purpose.

PROCEDURE

Procedure.

2. The sieve shall be weighed on an analytical balance, the weight being recorded in a figure carried to the third decimal place. The sieve shall then be wet on both sides with the liquid to be used for wash purposes.

3. The sample of the pigment to be tested shall be weighed. For most pigments 10 g. will be the proper quantity. For black pigments of low specific gravity 2 g. will be sufficient. For pigments like Prussian blue and graphite, 3 g. are generally used. The weighed samples of pigment shall then be transferred to the sieve.

If the pigment is one that is difficult to wet with the wash liquid, the quantity weighed out should first be placed in a beaker containing some of a liquid that easily wets the pigment and that is miscible with the wash liquid. For example, if water is to be used as the wash liquid, alcohol may be used as the wetting medium. The pigment is then gently stirred with the wetting liquid and the contents of the beaker transferred to the sieve. Where small particles of pigment

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

[2] See p. 917.

are retained on the stirring rod or walls of the beaker they may easily be removed with the brush.

4. The sieve shall then be held under a tap delivering about 300 to 500 cc. of wash liquid per minute. By slightly shaking the sieve, the pigment will be rapidly carried through. A soft camel's hair brush may be used in aiding the operation. If the sieve is held at a slight angle so that the pigment will gradually collect at one edge during the washing process, and then rotated, the pigment may be brushed out rapidly, with no risk of clogging the screen.

5. After the majority of the finely divided portion of the pigment has passed through the sieve (from 2 minutes to 1 hour, according to kind of pigment), the sieve shall be placed in an 8-in. porcelain dish containing 250 cc. of the wash liquid. The sieve will thus be covered to a depth of about $\frac{1}{2}$ in. The pigment remaining on the sieve shall be brushed with a soft 1-in. camel's hair brush at the rate of two strokes per second during two periods of 10 seconds each. The sieve shall then be raised from the dish after each 10-second period to let the liquid on the sieve run through. The liquid in the dish should be changed after every two brushing periods described above. This operation shall be continued until the wash liquid passing over the residue and through the sieve is clear and free from solid particles.

NOTE.—When the operator thinks he has completed the washing he should catch about 200 cc. of the liquid in a clean 400-cc. beaker, stir vigorously and set the beaker on a black surface in the case of white pigments and a white surface in the case of colored pigments. The washing is not complete until a test as described above fails to show any particles collected about the middle of the bottom of the beaker.

Occasionally pigments will be found that foam when water is used as the wash liquid. In such instances, during the last washing in the porcelain dish, the use of a liquid that breaks down the foaming and is readily miscible with water, such as alcohol, will usually overcome this difficulty.

6. The pigment particles adhering to the brush shall then be washed back onto the sieve and the water below the sieve wiped off. A few drops of alcohol and then of ether should be added to expedite drying. The screen shall be dried for one hour in an oven at 105° C., or on a radiator, cooled, weighed, and the percentage of coarse particles calculated.

7. For pigments requiring wash liquids such as turpentine, mineral spirits, or kerosine, the wash liquid shall be siphoned from a vessel onto the sieve, finishing with ether to remove the wash liquid adhering to the sieve.

STANDARD METHODS OF TESTING OLEO-RESINOUS VARNISHES[1]

Serial Designation: D 154 – 25

These methods are issued under the fixed designation D 154; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED IN AMENDED FORM, 1924; REVISED, 1925.

1. (*a*) All tests shall be made at room temperature between 21 and 32° C. (70 and 90° F.).

(*b*) All tests shall be made in diffused light (not in direct sunlight).

(*c*) When a can of varnish has been opened and part of its contents has been used, the remainder shall immediately be placed in air-tight containers which the varnish entirely fills, leaving not more than 2 per cent of air space.

(*d*) The tin panels used shall be cut from bright tin plate weighing not more than 25 g. nor less than 19 g. per square decimeter (0.51 to 0.39 lb. per sq. ft.).

NOTE.—Commercial No. 31 gage bright tin plate should weigh about 0.44 lb. per sq. ft.

It is important that the tin plate shall be within the limits prescribed. The panel shall be about 7.5 by 13 cm. (3 by 5 in.) and shall be thoroughly cleaned with benzol immediately before using.

NOTE.—It is important that the rags used in wiping the panels are clean.

APPEARANCE

Appearance.

2. Some of the thoroughly mixed sample shall be poured into a clear glass bottle or test tube, 1.5 to 2.0 cm. ($\frac{5}{8}$ to $\frac{13}{16}$ in.) in diameter to a depth of at least 2.5 cm. The varnish shall then be examined by transmitted light and shall be clear and transparent.

COLOR

Color.

3. (*a*) Standard color solutions shall be prepared by dissolving 1, 2, 3, 4, 5 and 6 g., respectively, of pure powdered potassium bichromate in 100 cc. of pure concentrated H_2SO_4 (sp. gr. 1.84). Gentle heat may be applied, if necessary, to effect the solution of bichromate.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

(*b*) The standard color solutions and a sample of the varnish to be tested shall be poured into thin-walled glass tubes 1.5 to 2.0 cm. ($\frac{5}{8}$ to $\frac{13}{16}$ in.) in diameter to a depth of at least 2.5 cm. The color comparison shall be made by placing the tubes close together and looking through them by transmitted light.

Since the potassium bichromate - sulfuric acid solution must be freshly made for this color comparison, it is frequently more convenient to compare samples with a series of permanently sealed tubes of varnish which have been previously found to be lighter in color than the standard solutions. When a sample is found to be darker than a standard tube of varnish, the bichromate standard should be made up for final decision. The color of the varnish shall be stated in terms of the standard (calling the standards No. 1, No. 2, No. 3, etc.) with which it is equal or lighter than.

NOTE.—Stabilized caramel solutions or other permanent colored liquids may be used as secondary standards.

NON-VOLATILE MATTER

Non-Volatile Matter.

4. A portion of the sample of the varnish shall be poured into a stoppered bottle or weighing pipette and weighed. About 1.5 g. of the sample shall be transferred to a flat-bottomed metal dish about 8 cm. in diameter. (Note.—A friction-top can plug is satisfactory.) The container shall be weighed again, and the exact weight of the portion of the sample transferred to the weighed dish shall be calculated by difference. The dish with its contents shall be heated for 3 hours in an oven maintained at 105 to 110° C. (221 to 230° F.). It shall then be weighed after cooling.

5. The ratio of the weight of the residue to that of the sample, expressed as a percentage, shall be taken as the percentage of non-volatile matter in the varnish.

ELASTICITY OR TOUGHNESS

Elasticity or Toughness.

6. The elasticity or toughness of the varnish shall be determined by proportionately reducing its elasticity or toughness by the addition of a standard solution of Run Kauri gum in pure spirits of turpentine.

Preparation of Standard Run Kauri.

7. (*a*) A distillation flask, water-cooled condenser and a tared receiver shall be arranged on a balance. Clear, bright, hard pieces of Kauri gum broken to the size of a pea shall be placed in the flask to about one-third its capacity. The gum shall be carefully melted and distilled until 25 per cent by weight is collected in the tared receiver. At the end of the distillation the thermometer in the distillation flask with the bulb at the level of the discharging end of the

flask should register about 316° C. (600° F.). The residue shall be poured into a clean pan and when cold it shall be broken into small pieces.

(*b*) *Standard Run Kauri Solution.*—A quantity of the small broken pieces of Run Kauri, together with twice its weight of freshly re-distilled spirits of turpentine, using only that portion distilling between 153 and 170° C. (308 and 338° F.), shall be placed in a carefully tared beaker and dissolved by heating to a temperature of about 149° C. (300° F.). It shall then be brought to correct weight when cooled by the addition of the amount of re-distilled spirits of turpentine necessary to replace the loss by evaporation during the dissolving of the gum.

Procedure.

8. (*a*) Having carefully determined the non-volatile content of the varnish in accordance with Section 4, 100 parts of the varnish by weight shall be taken and to it shall be added an amount of the standard Run Kauri solution equivalent to 50 per cent by weight of the non-volatile matter in the varnish and mixed thoroughly.

NOTE.—The 50-per-cent standard Run Kauri reduction is given to illustrate the method. Any other percentage of standard Run Kauri reduction may be used, depending on what is required of the particular sample being tested.

(*b*) The varnish shall be flowed upon one of the tin panels described in Section 1 (*d*) and the panel be permitted to stand in a nearly vertical position at room temperature for nearly one hour. The panel shall then be placed in a horizontal position in a properly ventilated oven and baked for five hours at from 95 to 100° C. The panels shall then be removed from the oven and permitted to cool at room temperature (preferably 24° C. (75° F.)) for 15 minutes.

Bending the Panel.

9. The panel shall be placed with the varnished side uppermost over a 3 mm. ($\frac{1}{8}$-in.) rod, held firmly by suitable supports, at a point equi-distant from the top and bottom edges of the panel and bent double rapidly. The varnish shall show no cracking whatsoever at the point of bending. For accurate results the bending of the panel should always be done at 24° C. (75° F.) for a lowering of the temperature will lower the percentage of reduction that the varnish will withstand without cracking, while an increase in temperature increases the percentage of reduction that the varnish will withstand.

10. Varnishes which do not show cracks under this test shall be reported as passing a fifty-per-cent reduction, while those that do crack shall be reported as not passing a fifty-per-cent reduction.

The varnishes which have not cracked shall be tested again, changing the amount of reduction to sixty per cent, and, if they pass this amount of reduction, they shall be reduced to seventy per cent.

In a similar manner, varnishes which have cracked at fifty per cent shall be tested, using reductions of thirty and forty per cent.

In this way the limits shall be determined within ten per cent at which a varnish passes one amount of reduction and does not pass the next. For example, varnishes may be reported as passing forty per cent, and breaking at fifty per cent.

NOTE.—It is suggested that a 20-g. sample of varnish shall be sufficient for each reduction. If the non-volatile content of the varnish should be 48.6 per cent, then 4.86 g. of standard Run Kauri solution should be added to the 20 g. of varnish and a fifty-per-cent reduction will be obtained.

FLASH POINT

11. The flash point shall be determined in accordance with the Standard Method of Test for Flash Point of Volatile Flammable Liquids (Serial Designation: D 56) of the American Society for Testing Materials.[1]

VISCOSITY

12. Viscosity shall be determined by comparison at 25° C. (77° F.) with secondary standards whose viscosity expressed in poises has been accurately determined at that temperature.

NOTE.—Gardner-Holdt tubes may be used. See *Circular No. 178*, Scientific Section, Paint Manufacturers' Association of the United States.

WATER TEST

13. The varnish shall be poured on one of the standard tin panels and allowed to drain in a nearly vertical position and dry for 48 hours. The panel shall be placed in a beaker containing about 7 cm. (2.5 in.) of distilled water at room temperature (immersing the end of the panel which was uppermost during the drying) and left in water for 18 hours. The panel shall then be removed from the water, wiped carefully, and allowed to dry out at room temperature. The time required for whitening, if any, to disappear, shall be noted. The results of the water test shall be reported as follows:

(*1*) Not visibly affected.

(*2*) Whitening disappears within 20 minuts.

(*3*) Whitening does not disappear in 20 minutes, but does disappear within 2 hours.

(*4*) Whitening does not disappear within 2 hours, but does disappear within 24 hours.

(*5*) Whitening does not disappear within 24 hours.

Blooming, which sometimes occurs on immersion, is considered as a degree of whitening.

[1] See p. 300.

STANDARD METHODS OF TESTING SHELLAC[1]

Serial Designation: D 29 - 25

These methods are issued under the fixed designation D 29; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1914; ADOPTED, 1917; REVISED, 1924, 1925.

DETERMINATION OF MATTER INSOLUBLE IN HOT ALCOHOL

(*a*) *Continuous Extraction Method* (suitable for all grades of lac).

APPARATUS

1. The extraction apparatus (Fig. 1) shall consist of a wide-neck flask in which is suspended a metal return condenser. From the lower part of this condenser is hung a siphon tube of Knoefler type. The extraction cartridge used for the determination shall be cut down to such a size that the top of the cartridge is just above the upper curve of the siphon. It shall be supported on three indentations in the glass so that there will be a little space beneath and around the cartridge to permit of a free flow of liquid.

NOTE.—Any type of siphon extractor where the siphon is continuously surrounded by the vapors of boiling alcohol may be substituted for the above form of apparatus.

SOLUTIONS REQUIRED

2. *95-per-cent Alcohol.*—Specially denatured alcohol, U. S. Internal Revenue Bureau Formula No. 1 or Formula No. 30.

METHOD

3. Prepare an extraction cartridge 26 mm. in diameter by 80 mm. in height (Schleicher and Schull No. 603 or the equivalent). Place the cartridge in the extraction apparatus, Fig. 1, and extract for 30 minutes with boiling 95-per-cent alcohol.

Dry in an air bath at 105° C., transfer to a glass-stoppered weighing bottle, cool and weigh. Dry to constant weight. A number of cartridges can be prepared and kept in glass-stoppered weighing

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

bottles until wanted. Now weigh accurately 5 g. of the lac in a 100-cc. beaker and dissolve in 75 cc. of boiling 95-per-cent alcohol by immersing the beaker in a hot water bath until all the shellac is dissolved and the wax is in solution. Transfer this solution quickly into the weighed extraction cartridge, previously wet with hot alcohol, putting the cartridge into a carbon filter tube of suitable size supported in a hot water bath (Fig. 2), the outlet tube extending through the bottom of the bath allowing the escape of the filtrate. Wash all the residue from the beaker into the cartridge with hot 95-per-cent alcohol. Put the

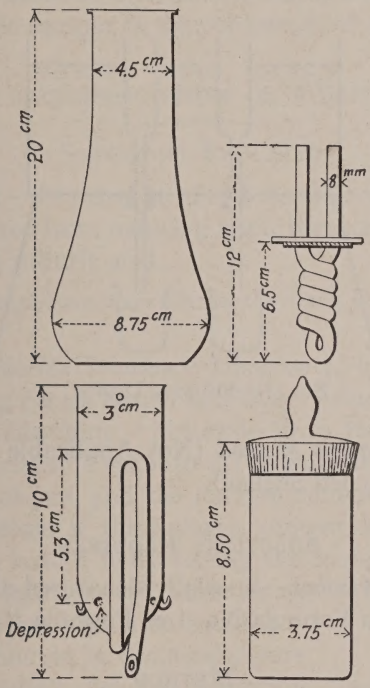

FIG. 1.—Extraction Apparatus for Alcohol-Insoluble Matter in Shellac

cartridge in the extraction apparatus (Fig. 1), and extract for exactly one hour.

Keep the alcohol boiling briskly during the extraction. The rate of extraction may be controlled by the use of an electric hot-point stove, 6 in. in diameter, and using the full current of 2.2 amperes at 110 volts. The volume of alcohol in the flask should be 125 cc. Protect the flask from drafts. Under these conditions the tube should siphon over at least 33 times in 1 hour. The condenser should be able to return all the alcohol volatilized during the vigorous boiling

of the contents of the flask, the object being to effect the maximum extraction during the specified time.

The weight of the residue insoluble in alcohol thus obtained, divided by the weight of the sample, and this quotient multiplied by 100 is the percentage of alcohol-insoluble matter in the lac.

NOTE.—When the determination of alcohol-insoluble matter in bleached shellac is required, the sample shall be dried if in the form of bars or hanks or ground shellac, as the water present dilutes the alcohol to a point where solution may not be complete. It is recommended that in preparing shellac for this determination, a separate portion be dried by exposure to the air in a thin layer, without the application of heat.

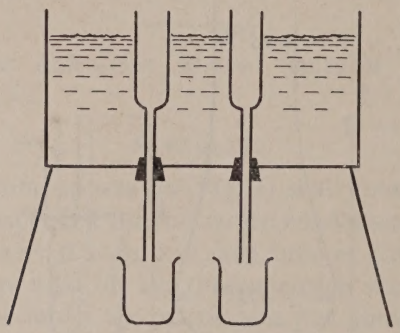

FIG. 2.—Filtering Device

(*b*) *Gooch Filtration Method* (Not Applicable for Stick, Seed or Grain Lac and Bleached Shellac).

SOLUTIONS REQUIRED

4. *95-per-cent Alcohol.*—Specially denatured alcohol, U. S. Internal Revenue Bureau Formula No. 1 or Formula No. 30.

METHOD

5. Grind a 50-g. sample fine enough to pass a No. 40 sieve. Weigh accurately 2 g. of the sample and transfer to a small beaker, heat the shellac with 25 cc. of 95-per-cent alcohol. Prepare a Gooch crucible with an asbestos pad in the usual manner[1] and dry it to constant weight. Arrange the crucible for filtration by suction and pour sufficient boiling alcohol through it to thoroughly heat the crucible.

NOTE.—A cold crucible will congeal the wax and prevent filtration.

[1] For the preparation of the Gooch crucible see any standard text on quantitative analysis, for example, Fresenius (Cohn Translation), p. 120, 1904, or Treadwell-Hall, third edition, Vol. II, p. 26.

Immediately filter the boiling solution through the crucible, using suction, transfer the insoluble material from the beaker to the crucible, using a "policeman" if necessary and a wash bottle containing hot alcohol until the washings are colorless and then wash five more times, nearly filling the crucible each time with boiling alcohol.

NOTE.—It will be necessary to shut off the suction momentarily to fill the crucible.

Wash off any film of shellac on the sides or bottom of the crucible with hot alcohol and dry to constant weight in an oven at 105 to 110° C. The weight of the residue in the crucible, multiplied by 100 and divided by the weight of the sample, is the percentage of material insoluble in hot alcohol.

DETERMINATION OF ROSIN

SOLUTIONS REQUIRED

6. *Acetic Acid.*—99-per-cent glacial acetic acid having a melting point of 14.8° C., free from reducing impurities as shown by its action on a bichromate in sulfuric acid.

NOTE.—If these requirements are not met the results of the rosin determination will be erratic.

Iodine Monochloride Solution.—Dissolve 13 g. of iodine in a liter of acetic acid, using gentle heat if necessary, determining the strength by titration with thiosulfate. Set aside 50 to 100 cc. of the solution and introduce dry chlorine gas into the remainder until the characteristic color change occurs, and the halogen content has been doubled. By titration, ascertain if the halogen content has been more than doubled, and if so reduce it by adding the requisite quantity of the iodine acetic acid solution. A slight excess of iodine does no harm but an excess of chlorine must be avoided.

Chloroform.—Should be chemically pure.

Sodium Thiosulfate Solution.—Dissolve 24.83 g. of the pure salt in a liter of cold, boiled, distilled water. Standardize by titrating against freshly resublimed iodine. It is recommended that the resublimed iodine be collected into a glass-stoppered weighing bottle and weighed after cooling.

Starch Solution.—Dissolve 0.2 g. of starch per 100 cc. of water and boil.

METHOD

7. Introduce 0.2 g. of ground shellac into a 250 cc. dry bottle of clear glass with a ground-glass stopper, add 20 cc. of glacial acetic acid and warm the mixture gently on top of a hot water bath until

solution is complete (except for the wax). A pure shellac is rather difficultly soluble; solution is quicker according to the proportion of rosin present. Add 10 cc. of chloroform and cool the solution to 21.5 to 22.5° C. The bottles should be allowed to stand half immersed in a shallow pan of water, well insulated or equipped with a suitable thermostat, at least 30 minutes at 21.5 or 22.5° C. before the Wijs solution is added. Add 20 cc. of Wijs solution (which shall be at a temperature of 21.5 to 22.5° C.) from a pipette, having a rather small delivery aperture (about 30 seconds). Close the bottle, place it back into the pan of water, and note the time. The bottles must be kept half immersed in water at 21.5 to 22.5° C. during the one hour that the shellac is exposed to the Wijs solution. Agitate the bottles occasionally during that hour.

NOTE.—If a number of samples are being run, at least 5 minutes should be allowed between the additions of the Wijs solution.

After exactly one hour, add 10 cc. of freshly prepared 10-per-cent potassium iodide water solution, washing into the bottle any Wijs solution on the stopper with the same. Titrate the solution immediately with the 0.1 *N* sodium thiosulfate solution, allowing the solution to run in slowly (about 25 to 30 cc.) with vigorous shaking until the solution becomes a straw color. Now add 15 cc. of freshly prepared starch solution and finish titrating. The end point is sharp, as the reaction products of shellac remain dissolved in the chloroform; any color returning after $\frac{1}{2}$ minute or so is disregarded.

A blank determination shall be run at the same time on the reagents. The blank is necessary on account of the well-known effect of temperature changes on the volume, and possibly loss of strength of the Wijs solution. A sample of pure shellac of known iodine value should also be run with every set of tests on unknown samples.

In the case of grossly adulterated samples, or in the testing of pure rosin, it is necessary to use, instead of 0.2 g. of material, a smaller amount (0.15 g. or 0.1 g.) in order that the excess of iodine monochloride may not be too greatly reduced, since the excess of halogen is one of the factors in determining the amount of absorption. In case less than 25 cc. of the thiosulfate solution are required, another test should be made, using a smaller amount of the shellac to be tested.

In weighing shellac, some difficulty is at times experienced on account of its electrical properties. In very dry weather it may be found that the necessary handling to prepare it for weighing has

electrified it, and that it may be necessary to leave it on the balance pan at rest for a few minutes before taking the final weight.

No pure shellacs show a higher iodine absorption than 18. As pure shellac is relatively a high-priced material and as the variation between its highest and lowest figures is not great, it is recommended that 18 should be taken as the standard figure for rosin-free shellac, determined by the method above described.

The determination for added rosin in bone-dry bleached shellac is run the same as above, except that the iodine absorption of pure bleached shellac is taken as 10 instead of 18.

It is recommended that the value for the iodine number of rosin be taken as 228. The results of using in this method the value of 18 as the iodine number of shellac and 228 as the number of rosin, may be that a slightly lower percentage of rosin, under some circumstances, will be found than that which is actually present.

The percentage of rosin shall be determined from the formula:

Iodine number of shellac..... 18
" " " rosin..... 228
" " " mixture..... x

$$\frac{(x - 18)}{(228 - 18)} \times 100 = \text{percentage of rosin}$$

Iodine number of bleached shellac..... 10
" " " rosin..... 228
" " " mixture..... x

$$\frac{(x - 10)}{(228 - 10)} \times 100 = \text{percentage of rosin}$$

DETERMINATION OF MOISTURE IN SHELLAC

SAMPLING

8. Both orange and bleached shellac give off volatile matter at temperatures approaching 100° C. Bleached shellac alters chemically at these temperatures, becoming less soluble in alcohol. For these reasons the usual method of determining water by heating in the air bath at 100 to 110° C. is not applicable in the analysis of shellac.

9. *Dry Bleached Shellac.*—In sampling dry bleached shellac, about 1 lb. should be taken from different parts of the barrel and finely ground by running through a coffee mill. No attempt shall be made to sieve it. It should be rapidly mixed and transferred to a mason jar provided with a screw cap and rubber ring seal. The jar

should be filled not more than two-thirds full, leaving room for a thorough mixing by shaking the contents. It should be kept in a cool place and tested as promptly as possible. If too warm, the shellac may tend to lump together, in which case the lumps must be broken up by shaking the bottle.

10. *Bars or Hanks.*—In sampling bars or hanks it is recommended that a whole hank be taken. It should be crushed and ground as rapidly as possible.

11. *Ground Bleached Shellac.*—Ground bleached shellac may be treated as above, bearing in mind that the large amount of moisture present makes rapid handling imperative.

METHOD

12. *Dry-Bleached Shellac.*—Weigh 5 g. of the finely ground sample in a flat-bottomed dish about 4 in. in diameter and place the dish in a well-ventilated air bath for three to six hours at 38 to 43° C. One or two electric light bulbs provide a convenient source of heat. The temperature should not be allowed to rise above 43° C.

NOTE.—With poorly ventilated ovens the drying may take much longer. Completeness of drying should be ascertained by continuing the treatment to constant weight.

13. *Hanks and Ground Bleached.*—Proceed as in Section 12, but with the exception that the dish and its contents be allowed to dry in the air, or in a sulfuric acid desiccator over night before it is placed in the air bath.

14. *Orange Shellac.*—Proceed as in the case of Section 12.

NOTE.—Dry-bleached shellac is also termed "bone dry," "kiln dry," "vac dry," "bleached shellac."

NOTES

Average commercial orange shellac contains not over 2 per cent of moisture.

Average commercial regular dry-bleached and dry-refined bleached shellac contain not more than 5 per cent of moisture.

Average commercial regular and refined bleached shellac as ground, hanks or bars contain not more than 25 per cent of moisture.

DETERMINATION OF WAX

Method

15. Dissolve 10 g. of dry shellac in 200 cc. of 190° proof specially denatured alcohol, formula No. 1 or No. 30, with agitation at a temperature of 24° C. ± 3° C.

Allow the solution to stand for several hours, preferably over night, in a tall cylindrical vessel, maintained at a temperature of 24° C. ±3° C., until the wax has settled to a small layer at the bottom of the container. Decant the clear solution through a 12.5-cm. folded filter paper, taking care not to disturb the wax layer. Finally, transfer the wax to the filter, using 25 cc. of the above alcohol at the prescribed temperature. Wash the container and the wax with four 15-cc. portions of the prescribed alcohol. The final washings should be colorless. Allow most of the alcohol to evaporate from the filter paper and dissolve the wax by pouring successive small portions of boiling chloroform (U. S. P.) through the paper. Also wash the container with boiling chloroform and pour on the filter.

Collect the filtrate, which should contain all of the wax, in a tared dish, evaporate the chloroform and dry to constant weight at 105° C. Weigh the wax residue and calculate the percentage of wax.

The removal of the wax from the filter paper can also be accomplished in any suitable continuous extraction apparatus.

In the case of shellac varnishes, dilute a weight of the sample, corresponding to 10 g. of non-volatile matter, with 200 cc. of alcohol and proceed exactly as described above.

NOTE.—Average commercial orange and dry-bleached shellac will contain about 4 to 5.5 per cent of wax. Average commercial dry-bleached refined shellac will be practically free from wax.

STANDARD METHOD OF TEST
FOR
FLASH POINT OF VOLATILE FLAMMABLE LIQUIDS[1]

Serial Designation: D 56 – 21

This method is issued under the fixed designation D 56; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1918; ADOPTED IN AMENDED FORM, 1919; REVISED, 1921.

This method was approved April 4, 1923, as "Tentative American Standard" by the American Engineering Standards Committee.

Tester to be Used.

1. (*a*) The Tag Closed Tester shall be used for all volatile flammable liquids flashing below 175° F. with the exception of products classed as Fuel Oil. Determination of the flash point of fuel oil by the Tag Tester is permissible, but the Pensky-Martens Tester, as described in the Standard Method of Test for Flash Point by Means of the Pensky-Martens Closed Tester (Serial Designation: D 93) of the American Society for Testing Materials,[2] is to be preferred.

Dimensions of Tester.

(*b*) The Tag Closed Tester shall conform to the following dimensions within the limits of tolerances given:

DIMENSION.	NORMAL.	TOLERANCE.
Depth of water surface below top of cup, in.	$1\frac{3}{32}$ (27.8 mm.)	$\pm\frac{1}{64}$ (0.4 mm.)
Depth of oil surface below top of cup, in.	$1\frac{5}{32}$ (29.4 mm.)	$\pm\frac{1}{64}$ (0.4 mm.)
Depth of top of bulb of oil thermometer when in place below top of cup, in.	$1\frac{5}{16}$ (33.3 mm.)	$\pm\frac{1}{32}$ (0.8 mm.)
Inside diameter of oil cup at top, in.	$2\frac{1}{8}$ (54.0 mm.)	±0.005 (0.1 mm.)
Weight of oil cup, g.	68	±1
Diameter of bead on top of cover, in.	$\frac{5}{32}$ (4.0 mm.)	$\pm\frac{1}{64}$ (0.4 mm.)

The plane of underside of cover to be between the top and bottom of the burner tip when the latter is fully depressed.

Specifications for Thermometer.

(*c*) The thermometer shall conform to the following requirements. These specifications cover a special thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being −7 to +110° C. or +20 to +230° F., respectively.

[1] Under the standardization procedure of the Society, this method is under the joint jurisdiction of the A.S.T.M. Committees D-1 on Preservative Coatings for Structural Materials and D-2 on Petroleum Products and Lubricants.

[2] See p. 395.

TYPE: Etched stem, glass.

LIQUID: Mercury.

RANGE AND SUBDIVISION: −7 to +110° C. in 0.5° C. or +20 to +230° F. in 1° F.

TOTAL LENGTH: 273 to 277 mm. (10.75 to 10.92 in.).

STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).

BULB: Corning normal or equally suitable thermometric glass.

Length, 9 to 13 mm. (0.35 to 0.51 in.).

Diameter, not greater than stem.

DISTANCE TO −7° C. OR +20° F. LINE FROM BOTTOM OF BULB: 75 to 90 mm. (2.93 to 3.54 in.).

DISTANCE TO 110° C. OR 230° F. LINE FROM TOP OF THERMOMETER: 25 to 40 mm. (0.98 to 1.57 in.).

EXPANSION CHAMBER: To permit heating the thermometer at least 50° C. (90° F.) above highest temperature on scale.

FILLING ABOVE MERCURY: Nitrogen gas.

TOP FINISH: Glass ring.

GRADUATION: All lines, figures, and letters clear cut and distinct. The whole degree Centigrade lines or the first and each succeeding 5° F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 5° C. or 10° F.

IMMERSION: 57 mm. or 2¼ in. The words "57-mm. immersion" on Centigrade or "2¼-in. immersion" on Fahrenheit thermometers and a line around the stem 57.0 mm. or 2.25 in. above the bottom of the bulb shall be etched on the thermometer.

SPECIAL MARKING:[1] "A.S.T.M. P.M. and Tag.," a serial number and the manufacturer's name or trade mark shall be etched on the stem.

SCALE ERROR: The error at any point of the scale when the thermometer is standardized as provided below shall not exceed 0.5° C. or 1° F., respectively.

STANDARDIZATION: The thermometer shall be standardized at the ice point and at intervals of approximately 30° C. or 50° F. for 57-mm. or 2¼-in. immersion and for the following temperatures of the emergent mercury column:

THERMOMETER READING	AVERAGE TEMPERATURE OF EMERGENT MERCURY COLUMN
20° C. 70° F.	20° C. 70° F.
40° C. 100° F.	31° C. 86° F.
70° C. 150° F.	40° C. 104° F.
100° C. 212° F.	48° C. 118° F.

CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. P.M. and Tag., −7 to +110° C." or "A.S.T.M. P.M. and Tag., +20 to +230° F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

[1] This thermometer is identical with the low range instrument specified for use with the A.S.T.M. Pensky-Martens Closed Tester and described in the Standard Method of Test for Flash Point by Means of the Pensky-Martens Closed Tester (Serial Designation: D 93) of the American Society for Testing Materials, p. 395.

General Directions.

2. (*a*) If gas is available, connect a $\frac{1}{8}$-in. rubber tube to the corrugated gas connection on the oil cup cover. If no gas is available, unscrew the test flame burner-tip from the oil chamber on the cover, and insert a wick of cotton cord in the burner-tip and replace it. Put a small quantity of cotton waste in the oil chamber, and insert a small quantity of signal, sperm or lard oil in the chamber, light the wick and adjust the flame, so that it is exactly the size of the small white bead mounted on the top of the tester.

(*b*) The test shall be performed in a dim light so that the flash may be seen plainly.

(*c*) Surround the tester on three sides with an inclosure to keep away draughts.

A shield about 18 in. square and 2 ft. high, open in front, is satisfactory, but any safe precaution against all possible room draughts is acceptable. Tests made in a laboratory hood or near ventilators will give unreliable results.

(*d*) See that the tester sets firm and level.

(*e*) For accuracy, the flash-point thermometers which are especially designed for the instrument should be used, as the position of the bulb of the thermometer in the oil cup is essential.

Water Bath.

3. Put the water-bath thermometer in place, and put a receptacle under the overflow spout to catch the overflow. Fill the water bath with water at such a temperature that, when testing is started, the temperature of the water bath will be at least 20° F. (11° C.) below the probable flash point of the oil to be tested.

Oil Cup.

4. Put the oil cup in place in the water bath. Measure 50 cc. of the oil to be tested in a pipette or a graduate, and place in the oil cup. The temperature of the oil shall be at least 20° F. (11° C.) below its probable flash point when testing is started. Destroy any bubbles on the surface of the oil. Put on the cover, with flash-point thermometer in place and gas tube attached. Light the pilot light on the cover and adjust the flame to the size of the small white bead on the cover.

Heating.

5. Light and place the heating lamp, filled with alcohol, in the base of the tester and see that it is centrally located. Adjust the flame of the alcohol lamp so that the temperature of the oil in the cup rises at the rate of about 1.8° F. (1° C.) per minute, not faster than 2° F. (1.1° C.) nor slower than 1.6° F. (0.9° C.) per minute.

Testing.

6. (*a*) Record the barometric pressure which, in the absence of a laboratory instrument, may be obtained from the nearest Weather Bureau Station.

(*b*) Record the temperature of the oil sample at start.

(*c*) When the temperature of the oil reaches 9° F. (5° C.) below the probable flash point of the oil, turn the knob on the cover so as to introduce the test flame into the cup, and turn it *promptly* back again. Do not let it snap back. *The time consumed in turning the knob down and back should be about one full second,* or the time required to pronounce distinctly the words "one-thousand-and-one."

(*d*) Record the time of making the first introduction of the test flame.

(*e*) Record the temperature of the oil sample at the time of first test.

(*f*) Repeat the application of the test flame at every 1° F. (0.5° C.) rise in temperature of the oil until there is a flash of the oil within the cup.

Do not be misled by an enlargement of the test flame or halo around it when it enters the cup, or by slight flickering of the flame; the true flash consumes the gas in the top of the cup and causes a very slight explosion.

(*g*) Record the time at which the flash point is reached.

(*h*) Record the flash point.

(*i*) If the rise in temperature of the oil, from the "time of making the first introduction of the test flame" to the "time at which the flash point is reached" was faster than 2° F. (1.1° C.) or slower than 1.6° F. (0.9° C.) per minute, the test should be questioned, and the alcohol heating lamp adjusted to correct the rate of heating. It will be found that the wick of this lamp can be so accurately adjusted as to give a uniform rate of rise in temperature within the above limits and remain so.

Repeat Tests.

7. (*a*) It is not necessary to turn off the test flame with the small regulating valve on the cover; leave it adjusted to give the proper size of flame.

(*b*) Having completed the preliminary test, remove the heating lamp, lift up the oil cup cover, and wipe off the thermometer bulb. Lift out the oil cup, and empty and carefully wipe it. Throw away all oil samples after once used in making a test.

(*c*) Pour cold water into the water bath, allowing it to overflow into a receptacle, until the temperature of the water in the bath is lowered to 15° F. (8° C.) below the flash point of the oil, as shown by the previous test.

With cold water of nearly constant temperature, it will be found that a uniform amount will be required to reduce the temperature of the water bath to the required point.

(*d*) Place the oil cup back in the bath and measure into it a 50-cc. charge of fresh oil. Destroy any bubbles on the surface of the oil, put

on the cover with its thermometer, put in the heating lamp, record the temperature of the oil, and proceed to repeat the test as described above in Sections 4 to 6, inclusive. Introduce the test flame for first time at a temperature of 10° F. (5.5° C.) below the flash point obtained on the previous test.

Average Value of Flash Point.

8. If two or more determinations agree within 1° F. (0.5° C.), the average of these results, corrected for barometric pressure, shall be considered the flash point. If two determinations do not check within 1° F. (0.5° C.), a third determination shall be made and, if the maximum variation of the three tests is not greater than 2° F. (1° C.), their average, after correcting for barometric pressure, shall be considered the flash point.

Correction for Barometric Pressure.

9. Correction for barometric pressure shall only be made in cases of dispute or when the barometer reading varies more than $\frac{1}{2}$ in. (13 mm.) from the standard pressure of 29.92 in. (760 mm.). When the barometer reading is below this standard pressure, add to the thermometer reading 1.6° F. (0.9° C.) for each inch (25 mm.) of barometer difference to obtain the true flash point. When the barometer reading is above the standard pressure, deduct 1.6° F. (0.9° C.) for each inch (25 mm.) of barometer difference to obtain the true flash point.

STANDARD METHODS OF ROUTINE ANALYSIS OF WHITE PIGMENTS[1]

Serial Designation: D 34 – 27

These methods are issued under the fixed designation D 34; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1915; ADOPTED, 1917; REVISED, 1927.

These methods were approved April 30, 1923, as "Tentative American Standard" by the American Engineering Standards Committee.

METHOD FOR VERY SMALL AMOUNTS OF IRON

Lead Pigments.—Treat sample with HNO_3 (1 : 1) in usual manner, dilute with H_2O, add H_2SO_4 to precipitate bulk of lead (not necessary to evaporate down); cool, filter, wash with 1 to 2 per cent of H_2SO_4, make filtrate just alkaline with NH_4OH, then just acid with dilute HNO_3, determine iron colorimetrically by the thiocyanate method, using same amounts of reagents in preparing standards. If sample contains insoluble matter, filter out and wash with hot water till Pb-free, and to filtrate add H_2SO_4 and proceed as given. The insoluble is ignited, treated with HF and H_2SO_4 in usual manner, brought into solution (filter out any $BaSO_4$), and added to filtrate from $PbSO_4$. If necessary, solution may be made up to volume and aliquots taken. Copper, if present, shall be removed by precipitating the Fe with NH_4OH, filtering, washing, redissolving $Fe(OH)_3$ and proceeding as above.

Other Pigments.—Treat as above, omitting the addition of H_2SO_4.

GENERAL METHOD

True specific gravity shall be determined in accordance with the Standard Methods of Test for Specific Gravity of Pigments (Serial Designation: D 153) of the American Society for Testing Materials.[2]

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

[2] See p. 277.

BASIC CARBONATE OF LEAD

Total Lead (*Gravimetric*).—Dissolve 1 g. in 20 cc. of HNO_3 (1:1) in a covered beaker, heating till all CO_2 is expelled; wash off cover, add 20 cc. of H_2SO_4 (1:1) and evaporate to fumes of SO_3, cool, add about 150 cc. of water and 150 cc. of ethyl alcohol; let stand in *cold* water 1 hour, filter on a Gooch crucible, wash with 95-per-cent ethyl alcohol, dry at 110° C., and weigh $PbSO_4$; calculate to PbO or to basic carbonate.[1] Instead of determining the lead as sulfate, the sample may be dissolved by boiling with acetic acid; then dilute to about 200 cc. with water, make alkaline with NH_4OH, then acid with acetic acid, heat to boiling and add 10 to 15 cc. of a 10-per-cent solution of potassium dichromate; heat till the yellow precipitate assumes an orange color. Let settle and filter on a Gooch crucible, washing by decantation with hot water till the washings are colorless finally transferring all of the precipitate. Then wash with 95-per-cent ethyl alcohol and then ether; dry at 110° C. and weigh $PbCrO_4$. (Any insoluble matter should be filtered out before precipitating the lead.)

Total Lead (*Volumetric*).—Dissolve 0.5 g. of sample in 10 cc. of concentrated hydrochloric acid, boil till solution is effected, cool, dilute to 40 cc., neutralize with ammonium hydroxide. Add acetic acid until distinctly acid, dilute to 200 cc. with hot water, boil and titrate with ammonium molybdate as follows:

Dissolve 4.25 g. of ammonium molybdate in water and make up to one liter. To standardize this solution, dissolve about 0.2 g. of pure lead foil in nitric acid (pure PbO or $PbSO_4$ may also be used), evaporate nearly to dryness, add 30 cc. of water, then 5 cc. H_2SO_4 (sp. gr. 1.84), cool, and filter. Drop filter with $PbSO_4$ into a flask, add 10 cc. of concentrated HCl, boil till completely disintegrated, add 15 cc. of HCl, 25 cc. of water, and NH_4OH till alkaline. Acidify with acetic acid, dilute to 200 cc. with hot water and boil. Titrate, using an outside indicator of one part of tannic acid in 300 parts of water.

It should be noted that when calcium is present, it forms a more or less insoluble molybdate, and results are apt to be high. With samples containing less than 10 per cent of lead, the lead should be precipitated as $PbSO_4$, filtered, redissolved and titrated as in the process of standardizing.

[1] This method of weighing lead sulfate is not accurate in the presence of calcium compounds.

Carbon Dioxide.—Determine by evolution with dilute hydrochloric acid absorbing in soda-lime or KOH solution. Calculate CO_2 to $PbCO_3$, subtract PbO equivalent from total PbO and calculate residual PbO to $Pb(OH)_2$.

Acetic Acid.[1]—Place 18 g. of the pigment in a 500-cc. flask, add 40 cc. of sirupy phosphoric acid, 18 g. of zinc dust and 50 cc. of water. Connect to a straight Liebig condenser, apply heat and distill down to a small bulk. Then pass steam into the flask until it becomes about half full of condensed water, shut off the steam and distill down to a small bulk—this operation being conducted twice. To the total distillate which was collected in a larger flask add 1 cc. of sirupy phosphoric acid, connect to a Liebig condenser, using a spray trap, and distill to a small volume—about 20 cc. Pass steam through till about 200 cc. of water condense in the distillation flask, shut off steam and continue the distillation. These operations of direct and steam distillations are conducted until 10 cc. of the distillate require only one drop of 0.1 *N* alkali to give a change in the presence of phenolphthalein. Then titrate the total distillate with 0.1 *N* sodium hydroxide and phenolphthalein and calculate the total acidity as acetic acid. It will be found convenient to titrate each 200-cc. portion of the distillate as collected.

Metallic Lead.[1]—Weigh 50 g. of the sample into a 400-cc. beaker, add a little water and add slowly 60 cc. of 40-per-cent acetic acid and after effervescence has ceased, boil on hot plate. Fill the beaker with water, let settle, and decant the clear solution. To the residue add 100 cc. of a mixture of 360 cc. of strong NH_4OH, 1080 cc. of water, 2160 cc. of 80-per-cent acetic acid and boil until all solution is complete. Fill beaker with water, let settle and decant the clear solution. Collect residue on a watch glass, floating off everything but metallic lead. Dry and weigh. Result $\times$ 2= percentage of metallic lead in sample.

The following method of A. N. Finn (unpublished) gives total basicity of a pure white lead: Place 2 g. of pigment in an evolution flask, add a little CO_2-free water, connect up to the separatory funnel and condenser (Knorr type), add through the funnel, finally washing down, 100 cc. of 0.25 *N* nitric acid, boil and absorb the CO_2 in soda-lime tube in usual manner (having H_2SO_4 and $CaCl_2$ drying tubes in train) and weigh. To the solution in the evolution flask, add about 20 cc. of neutral sodium-sulfate solution and titrate with 0.25 *N* sodium-hydroxide

[1] Thompson's Method, *Journal*, Soc. Chem. Ind., Vol. 24, p. 487 (1905)

solution (carbonate-free), using phenolphthalein. CO_2 is calculated to $PbCO_3$. The amount of 0.25 *N* acid corresponding to the CO_2 is calculated and deducted from the total amount of 0.25 *N* acid neutralized by the sample and the difference calculated to combined H_2O, from which $Pb(OH)_2$ is computed.

BASIC SULFATE OF LEAD.[1]

Determination of Total Lead.—Dissolve 1 g. of the sample in 100 cc. of a mixture consisting of 125 cc. of 80-per-cent acetic acid, 95 cc. of NH_4OH (sp. gr. 0.90) and 100 cc. of water. Add the solution while hot and dilute with about 50 cc. of water, boil until sample is dissolved. Dilute to 200 cc. and titrate with standard ammonium-molybdate solution as described under basic carbonate of lead. The ammonium-molybdate solution contains about 8.67 g. per liter and is standardized against pure lead foil, pure PbO or pure $PbSO_4$.

Determination of Total Zinc.—Boil 1 g. of the sample with 30 cc. of water, 4 g. of NH_4Cl and 6 cc. of concentrated HCl (some $PbSO_4$ or $PbCl_2$ may not dissolve). Dilute to 200 cc. with hot water, add 2 cc. of a saturated solution of sodium thiosulfate and titrate with a standard solution of potassium ferrocyanide in usual manner. Calculate the zinc to ZnO,[2] as follows:

99.70^a − percentage of ZnO found = percentage of lead constituents;

then

$$\frac{\left(\frac{\text{Molecular weight } PbSO_4}{\text{Atomic weight Pb}} \times \text{Percentage of Pb found}\right) - \text{Percentage of Pb constituents}}{\text{Molecular weight } PbSO_4 - \text{Molecular weight PbO}} = \text{percentage of PbO present.}$$

$$\frac{\left(\frac{\text{Molecular weight PbO}}{\text{Atomic weight Pb}} \times \text{Percentage of Pb found}\right) - \text{Percentage of Pb constituents}}{\text{Molecular weight PbO} - \text{Molecular weight } PbSO_4} = \text{percentage of } PbSO_4 \text{ present.}$$

Sulfur Dioxide.—Digest 2 g. of the sample with frequent stirring in 100 cc. of freshly boiled *cold* water and 5 cc. of concentrated HCl; let stand 10 to 15 minutes, add an excess of 0.01-normal iodine solution and titrate back with 0.01-normal sodium-thiosulfate solution, using starch indicator. Report as SO_2.

[1] J. A. Schaeffer's Method, *Journal of Industrial and Engineering Chemistry*, Vol. 6, p. 200 (1914), may be used.

[2] It would probably be more accurate to remove total lead as $PbSO_4$ and titrate the zinc in the filtrate after adding NH_4OH and HCl in usual manner.

[a] This figure (99.70) is the average total percentage of $PbSO_4$, PbO and ZnO, found in 270 total analyses of basic lead sulfate.

Run blank on reagents and make corrections. Or, the SO_2 may be titrated directly with iodine.

Soluble Zinc Sulfate.—Determine as under "Zinc-Lead and Leaded Zincs."

ZINC-LEAD AND LEADED-ZINCS (OZLO WHITE).

Total Lead and Zinc.—Dissolve 1 g. of the sample by boiling with 250 cc. of water and 20 cc. of concentrated HNO_3, add 5 cc. of concentrated H_2SO_4, and evaporate to copious fumes of SO_3; cool, add 250 cc. of water, let stand *cold* 1 to 2 hours, filter on Gooch crucible, wash with 1-per-cent H_2SO_4, ignite, and weigh as $PbSO_4$. Report as $PbSO_4$. The $PbSO_4$ may be filtered on paper and then dissolved and titrated with ammonium-molybdate solution as described under basic carbonate of lead. Make filtrate from $PbSO_4$ alkaline with NH_4OH, then acid with HCl, add 3 cc. of concentrated HCl, heat to nearly boiling, and titrate the total zinc with potassium-ferrocyanide solution using uranium-acetate solution as an outside indicator and calculate to ZnO. (Iron, copper, or other interfering substances should first be removed as described by Low.)

Sulfates other than Barium Sulfate.—Treat 0.5 g. of the sample with 5 cc. of water, 3 g. of NH_4Cl and 5 cc. of HCl saturated with bromine; digest (covered) on steam bath about 15 minutes, add 25 cc. of water, neutralize with dry Na_2CO_3, and add about 2 g. more, boil 10 to 15 minutes; let settle, dilute with hot water, filter and wash with hot water; redissolve in HCl, reprecipitate as above and wash thoroughly with hot water; acidify united filtrates with HCl, adding a slight excess; boil and add slight excess of 10-per-cent barium-chloride solution. Let stand on steam bath 1 hour, filter, wash with hot water, ignite, and weigh $BaSO_4$. Calculate to SO_3 (includes SO_3 formed from SO_2). Or, dissolve 0.5 g. of the sample in 25 cc. of water, 10 cc. of NH_4OH (sp. gr. 0.90) and HCl in slight excess; dilute to about 150 cc. with water and add a piece of aluminum foil which should about cover the bottom of the beaker—this held on bottom by means of a stirring rod. Heat gently till all lead is precipitated, decant through a filter, pressing the lead sponge with a flattened rod, and washing with hot water. Add to the filtrate a little pure bromine water, boil until bromine is expelled, add 15 cc. of 10-per-cent barium-chloride solution, let stand on steam bath one hour, filter, wash with hot water, ignite and

weigh as $BaSO_4$ (any $SrSO_4$ present is not decomposed in this method).[1]

Soluble Zinc Sulfate.—Heat nearly to boiling 2 g. of the sample with 150 cc. of water and 50 cc. of 95-per-cent alcohol for 30 minutes, filter, and wash with a mixture of alcohol and water (1:3). Heat filtrate to boiling and expel most of the alcohol; then determine SO_3 by usual method of precipitation with $BaCl_2$. Calculate to $ZnSO_4$ and to SO_3.

NOTE.—If sample contains Ca or Mg, the Pb and Zn should be separated by precipitation with H_2S after dissolving in HCl, making alkaline with NH_4OH and acid with acetic acid. The PbS+ZnS is dissolved in dilute HNO_3 and the Pb and Zn determined as above.

Sulfur Dioxide.—Determine as under "Basic Sulfate of Lead" or "Zinc Oxide."

ZINC OXIDE.

Total Zinc.—Dissolve 0.25 to 0.3 g. in 10 cc. of concentrated HCl and 20 cc. of H_2O, make alkaline with NH_4OH, then acid with HCl, add 3 cc. more of concentrated HCl, dilute to about 250 cc. with H_2O, heat nearly to boiling and titrate with standard potassium-ferrocyanide solution as described by Low.[2] Report as ZnO (includes Cd). Iron, copper or other interfering substances should be first removed as described by Low.

Total Soluble Sulfur.[3]—Moisten a 10-g. sample with water, add a few drops of bromine and then concentrated HCl, boil to expel bromine, filter from any insoluble and wash with hot water. Make alkaline with NH_4OH, then just slightly acid with HCl, heat to boiling and add about 15 cc. of hot barium-chloride solution. Let stand several hours (over night), filter on a weighed Gooch crucible, wash well with hot water, dry, ignite for five minutes, cool and weigh as $BaSO_4$. Calculate to S.

Sulfur Dioxide.[3]—Mix 5 g. of sample with 50 cc. of warm (freshly boiled and then partly cooled) water to an emulsion and pour into a glass-stoppered flask containing 18 cc. of HCl and exactly 25 cc. of 0.1-normal iodine solution, stopper and shake until all the oxide is dissolved. Titrate the excess of iodine as rapidly as possible with 0.1-normal sodium-thiosulfate solution. Calculate to SO_2.

Soluble Zinc Sulfate.—Determine as under "Zinc-Lead and Leaded-Zincs."

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[1] The solubility of $BaSO_4$ is increased by the presence of aluminium chloride. See J. W. Mellor. "A Treatise on Quantitative Inorganic Analysis," p. 615.

[2] Low, "Technical Methods of Ore Analysis."

[3] Method of G. Rigg.

LITHOPONE.

PONOLITH, JERSEY LILY WHITE, BECTON WHITE, CHARLTON WHITE, ORR'S WHITE.

Insoluble and Total Zinc.—Take 1 g. of the sample in a 200-cc. beaker, add 10 cc. of concentrated HCl, mix, and add in small portions about 1 g. of $KClO_3$, then heat on the steam bath until about half of the liquid is evaporated. Dilute with H_2O, add 5 cc. of dilute H_2SO_4 (1:10); boil, let settle, filter, wash, ignite, cool, and weigh the insoluble which should be only $BaSO_4$; make a qualitative examination for alumina and silica. The insoluble should be examined under the microscope for the presence of natural crystallin barytes. Sample may also be examined direct. Make filtrate from insoluble alkaline with NH_4OH, acid with HCl, add 3 cc. of concentrated HCl, dilute to about 250 cc. with H_2O, heat nearly to boiling and titrate with $K_4Fe(CN)_6$ solution as under zinc white. Calculate to Zn.

Zinc Oxide.—Treat a 4-g. sample of the lithopone for 4 hours with 200 cc. of 1-per-cent acetic acid at room temperature, stirring occasionally. Filter by suction on a double filter paper and wash with cold water; add to the clear filtrate 13 cc. of concentrated NH_4OH, neutralize with HCl and then add 3 cc. of concentrated HCl in excess. Heat to boiling and titrate with $K_4Fe(CN)_6$, using uranium-acetate solution as an outside indicator. Calculate to ZnO. Calculate this result to Zn, subtract from total Zn, and calculate the difference to ZnS. (Any $ZnCO_3$ or $ZnSO_4$ is included in the ZnO.)

Zinc Sulfide.[1]—Place 0.5 g. of pigment in evolution flask with about 10 g. of "feathered" or mossy zinc, add 50 cc. of water; insert the stopper carrying a separatory funnel and an exit tube. Run in 50 cc. of concentrated HCl from the funnel, having previously connected the exit tube to two absorption flasks in series; first flask contains 100 cc. of alkaline lead-nitrate solution, second flask, 50 cc. of same as a safety device. After all of the acid has run into the evolution flask, heat slowly, finally boiling until the first appearance of steam in the first absorption flask; disconnect, let the lead sulfide settle, filter, wash with cold water, then with hot water till neutral to litmus paper and washings give no test for lead. The PbS precipitate is dissolved in hot, dilute HNO_3, evaporated to fumes with

[1] Evolution Method of W. G. Scott, "White Paints and Painting Material," p. 257; see also Blair, "Chemical Analysis of Iron."

H_2SO_4 and finally weighed as $PbSO_4$. Calculate PbS or $PbSO_4$ to ZnS.

The alkaline lead solution is made as follows: Into 100 cc. of KOH solution (56 g. in 140 cc. of H_2O) pour a saturated solution of lead nitrate (250 g. in 500 cc. of H_2O) until the precipitate ceases to redissolve, stirring constantly while mixing. About 3 volumes of the lead solution will be required for one of the alkali.

Instead of absorbing the evolved H_2S in alkaline lead-nitrate solution, a solution of 8 g. of cadmium chloride in 250 cc. of water and 150 cc. of NH_4OH (sp. gr. 0.90) may be used. The CdS precipitate may be filtered on a weighed Gooch, washed with water containing a little NH_4OH, dried at 100° C., and weighed. Calculate to ZnS. It is better to filter the CdS on a small filter and wash as above, then place filter and precipitate in a beaker and dissolve in HCl and $KClO_3$ (keeping at room temperature at first), filter out any paper pulp or insoluble matter; make filtrate alkaline with NH_4OH, then just acid with HCl, heat to boiling and precipitate with $BaCl_2$ in usual manner. Filter, wash, ignite, and weigh $BaSO_4$. Calculate to ZnS.

For very rapid work the contents of the absorption flask, after all H_2S has been absorbed, may be washed into a vessel with *cold* water and diluted to about one liter, acidified with concentrated HCl and titrated with standard iodine solution, using starch indicator. (The precipitate should be completely dissolved.) The iodine solution is prepared by dissolving about 12.7 g. of pure resublimed iodine and 18 g. of KI in a little water and then diluting to one liter.

CALCIUM PIGMENTS.

WHITING, PARIS WHITE, SPANISH WHITE, AND CHALK.

Make a qualitative examination first; should contain only small amounts of insoluble matter (siliceous), iron, aluminum, sulfur, water or magnesium, and should be approximately 95 per cent $CaCO_3$.

Total Soluble Lime.[1]—Weigh out 0.75 g. of the pigment into a small crucible, ignite cautiously to dull redness to destroy organic matter, cool, transfer to a 400-cc. beaker, add 20 cc. of H_2O, cover, then add 15 cc. of concentrated HCl and 3 or 4 drops

[1] Meade, "Portland Cement."

of concentrated HNO_3, and boil till all the soluble matter is dissolved and all the CO_2 expelled. Wash off and remove the cover, dilute to about 150 cc. with freshly boiled H_2O, heat to boiling and add dilute NH_4OH (sp. gr. 0.96) carefully until a *slight* permanent precipitate forms. Heat to boiling and add 10 cc. of a 10-per-cent solution of oxalic acid; stir until the oxides of iron and aluminum are entirely dissolved and only a slight precipitate of calcium oxalate remains. Now add 200 cc. of boiling H_2O and sufficient saturated solution of ammonium oxalate (20 to 25 cc.) to precipitate the lime. Boil and stir for a few moments, remove from the heat, let settle and filter on an 11-cm. filter. Wash 10 times with 10-to-15-cc. portions of hot water. Place beaker in which precipitation was made under the funnel, pierce apex of filter with stirring rod and wash precipitate into beaker with hot water, pour warm dilute H_2SO_4 (1:4) through paper and wash a few times; add about 30 cc. of the dilute H_2SO_4 (1:4), dilute to about 250 cc., heat to 90° C. and titrate at once with standard $KMnO_4$ solution (solution should not be below 60° C. when end-point is reached). The $KMnO_4$ is best standardized against Bureau of Standards sodium oxalate.[1] Calculate to CaO and $CaCO_3$.

Mixed Calcium and Magnesium Carbonates.[2]—Weigh 1 g. of the finely powdered sample into a small porcelain dish, add 25 cc. of normal HCl, cover with a watch glass, and when effervescence has ceased, heat to boiling. Cool and titrate with normal NaOH solution, using methyl orange as indicator.

The calculation is as follows:

One gram CaO = 35.7 cc. of normal acid. CaO × 1.784 = $CaCO_3$. Subtract number of cubic centimeters of NaOH required from 25, result gives number of cubic centimeters of normal acid corresponding to the $CaCO_3$+$MgCO_3$. Multiply the weight of CaO in 1 g. of sample (as found in preceding section on total soluble lime) by 35.7; product gives number of cubic centimeters of normal acid corresponding to the CaO present; subtract from total number of cubic centimeters of acid required by $CaCO_3$+$MgCO_3$ and multiply result by 0.042, obtaining weight of $MgCO_3$ in 1 g. of sample. The $MgCO_3$ determined by this process should not differ more than 0.25 per cent from that obtained by more elaborate methods. It is to be noted that this method is a measure of the total alkalinity,

[1] *Circular No. 40*, Bureau of Standards.

[2] J. W. Mellor, "A Treatise on Quantitative Inorganic Analysis," p. 522.

and if Ca or Mg are present in other forms than carbonate, a complete analysis would be necessary to give percentages of $CaCO_3$ and $MgCO_3$.

Instead of the above, Newberry's[1] method for the simultaneous determination of calcium and magnesium carbonates may be used.

GYPSUM, TERRA ALBA, PLASTER OF PARIS.

Combined Water and Moisture.—Heat 1 g. of the sample in a covered porcelain crucible on an asbestos plate for 15 minutes, then heat bottom of crucible *dull red* for 10 minutes over a Bunsen burner, remove cover and heat for 30 to 40 minutes at a slightly lower temperature. Cool and weigh rapidly. Repeat to constant weight.

Combined water and moisture may also be determined by heating in an air bath at 200° C. to constant weight.

Insoluble Matter.—Boil 2 to 3 g. of the sample with 20 cc. of concentrated HCl, a few drops of HNO_3, and about 50 cc. of water; evaporate to dryness, boil residue repeatedly with 10-per-cent HCl, decanting through a filter; finally transfer to the filter, wash with hot water, ignite and weigh insoluble matter; test for $BaSO_4$. Make a qualitative examination of the filtrate from the insoluble matter to determine if Al, Fe, or Mg are present in abnormal amounts (should be mainly Ca and SO_3). Test a separate portion of the sample for CO_2; if desired determine as under "Basic Carbonate of Lead."

BARIUM PIGMENTS.

BARYTES OR BARITE, "BLANC FIXE."

Examine the sample microscopically to determine uniformity of grinding, size and angularity of particles, crystallin or amorphous. The sample should be at least 95 per cent $BaSO_4$.

Loss on Ignition.—Ignite 1 g. of the sample for 30 minutes. Loss may be due to organic matter, moisture, combined water and CO_2 (and SO_3 from $CaSO_4$ if present). If loss is appreciable, test for CO_2.

Soluble and Insoluble Matter.—Boil 1 g. with HCl (1:3), filter, wash with hot water, ignite, and weigh insoluble matter.

[1] Meade, "Portland Cement;" Mellor, "A Treatise on Quantitative Inorganic Analysis," p. 522. *Cement Engineering News*, Vol. 15, p. 35 (1903).

This may be treated with H_2SO_4 and HF in usual manner for SiO_2. In the absence of Al_2O_3 (from silicates) residue is considered as $BaSO_4$. Make qualitative tests of filtrate from the insoluble for Al, Fe, Ca, Mg, SO_3. Test a separate portion of sample qualitatively for CO_2.

SILICA PIGMENTS.

SILICA OR SILEX.

Silica or silex should be practically pure SiO_2. A qualitative examination will suffice in most cases with a determination of the loss on treating 1 g. of sample with H_2SO_4 and HF in usual manner.

CHINA CLAY AND ASBESTINE.

A qualitative analysis of China clay to determine if essentially hydrous aluminium silicate is generally all of the chemical tests necessary. Color, fineness, etc., are more important than analysis.

Asbestine is tested qualitatively to prove that material is as represented. Color, fineness, etc., are important. Asbestine should be examined under the microscope to note whether it is fibrous or talcose and whether silica or China clay are admixed. Samples of known origin should be used here as a guide for comparison.

STANDARD METHODS OF ROUTINE ANALYSIS OF DRY RED LEAD[1]

Serial Designation: D 49 - 27

These methods are issued under the fixed designation D 49; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1917; ADOPTED, 1918; REVISED, 1927.

These methods were approved April 30, 1923, as "Tentative American Standard" by the American Engineering Standards Committee.

1. The approximate formula of Red Lead is Pb_3O_4 (probably $PbO_2 \cdot 2PbO$).

SPECIFIC GRAVITY.

Specific Gravity.

2. True specific gravity shall be determined in accordance with the Standard Methods of Test for Specific Gravity of Pigments (Serial Designation: D 153) of the American Society for Testing Materials.[2]

MOISTURE.

Moisture.

3. Dry 2 g. of the sample for 2 hours at 105° C. The loss in weight is considered as moisture.

ORGANIC COLOR.

Organic Color.

4. Boil 2 g. of the sample with 25 cc. of 95-per-cent ethyl alcohol, let settle, decant off the supernatant liquid; boil residue with water, decant as before and boil residue with very dilute NH_4OH. If either the alcohol, water or NH_4OH is colored, organic coloring matter is indicated.

TOTAL LEAD AND INSOLUBLE MATTER.

Total Lead and Insoluble Matter.

5. Treat 1 g. of the sample with 15 cc. of HNO_3 (1 : 1) and sufficient H_2O_2 to dissolve all PbO_2 on warming. If any insoluble matter is present, add 25 cc. of water, boil, filter and wash with hot water. The insoluble matter contains free SiO_2 and should be examined for $BaSO_4$ and silicates, if appreciable. To the

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

[2] See p. 277.

original solution or filtrate from insoluble, add 20 cc. of concentrated H_2SO_4 and evaporate to SO_3 fumes; cool, add 150 cc. of water and 150 cc. of 95-per-cent ethyl alcohol, let stand *cold* 2 hours, filter on a Gooch crucible, wash with 95-per-cent alcohol, dry at 105 to 110° C. and weigh as $PbSO_4$. Calculate to PbO. Red lead is rarely adulterated, but should sample contain soluble barium compounds, the $PbSO_4$ obtained above will contain $BaSO_4$. In this case, digest above precipitate with acid ammonium-acetate solution, filter off $BaSO_4$, wash, ignite and weigh $BaSO_4$. Calculate to BaO or $BaCO_3$. In filtrate, determine the lead as $PbSO_4$ or $PbCrO_4$. If sample contains significant amounts of calcium or magnesium, the HNO_3–H_2O_2 solution is boiled till all lead is converted into nitrate and then the lead is determined as $PbCrO_4$.

If calcium and magnesium are to be determined, proceed as follows: Precipitate the lead as sulfide from a slightly acid (HCl) solution, dissolve the PbS in hot dilute HNO_3 and determine the lead as sulfate. Boil the filtrates from the PbS to expel H_2S, add a little bromine water to oxidize iron (if present), boil to expel bromine, and then add NH_4OH in slight excess. Filter off any precipitate of $Fe(OH)_3+Al(OH)_3$; wash with hot water. (If appreciable, redissolve in hot dilute HCl and reprecipitate with NH_4OH, ignite and weigh $Fe_2O_3+Al_2O_3$.) Manganese, if present, can be precipitated by adding bromine and NH_4OH and warming; filter, wash with hot water, ignite and weigh as Mn_3O_4. Unite all of the filtrates, make slightly acid with acetic acid, heat to boiling and pass H_2S into the hot solution till saturated (20 to 30 minutes); add 5 g. of NH_4Cl and let stand 5 hours; filter off any ZnS, wash with H_2S water, dissolve the ZnS in hot dilute HCl and determine the zinc by titration with $K_4Fe(CN)_6$. Or, boil off the H_2S, filter out any separated sulfur and determine the zinc as $Zn_2P_2O_7$. Calcium may be determined in the filtrate from the ZnS by expelling H_2S and then adding NH_4OH and ammonium oxalate in the usual manner. Titrate with $KMnO_4$. In the filtrate from calcium determine magnesium in the usual manner by precipitating with sodium-phosphate solution, finally weighing as $Mg_2P_2O_7$.

LEAD PEROXIDE (PbO_2) AND TRUE RED LEAD (Pb_3O_4).

(Method of Diehl[1] modified by Topf[2]—not applicable when substances are present, other than oxides of lead, that liberate iodine under conditions given.)

[1] *Dingl. polyt. Jour.*, Vol. 246, p. 196.
[2] *Zeitschrift für analytische Chemie*, Vol. 26, p. 296.

Lead Peroxide and True Red Lead.

6. Weigh 1 g. of finely ground sample into a 200-cc. Erlenmeyer flask, add a few drops of distilled water and rub the mixture to a smooth paste with a glass rod flattened on end. Mix in a small beaker 30 g. of c. p. "Tested Purity" crystallized sodium acetate, 2.4 g. of c. p. KI, 10 cc. of water and 10 cc. of 50-per-cent acetic acid; stir until all is liquid, warming gently; if necessary add 2 to 3 cc. of water, cool to room temperature and pour into the flask containing the red lead. Rub with the glass rod until nearly all the red lead has been dissolved; add 30 cc. of water containing 5 or 6 g. of sodium acetate, and titrate at once with decinormal sodium thiosulfate, adding the latter rather slowly and keeping the liquid constantly in motion by whirling the flask. When the solution has become light yellow, rub any undissolved particles up with the rod until free iodine no longer forms, wash off rod, add the sodium-thiosulfate solution until *pale yellow*, add starch solution and titrate until colorless, add decinormal iodine solution until blue color is just restored and subtract the amount used from the volume of sodium thiosulfate that had been added.

Calculation.—The iodine value of the sodium-thiosulfate solution multiplied by $0.942 = PbO_2$; the iodine value multiplied by $2.7 = Pb_3O_4$; the PbO_2 value multiplied by $2.86616 = Pb_3O_4$.

The sodium-thiosulfate solution and the starch solution shall be prepared as follows:

Sodium-Thiosulfate Solution (*decinormal*).—Dissolve 24.83 g. of c. p. sodium thiosulfate, freshly pulverized and dried between filter paper, and dilute with water to 1 liter at the temperature at which the titrations are to be made. The solution is best made with well-boiled water free from CO_2, or let stand 8 to 14 days before standardizing. Standardize with pure, re-sublimed iodine, as described in Treadwell-Hall, "Analytical Chemistry," Vol. II, p. 602 (1910), and also against pure potassium iodate; the two methods of standardization should agree within 0.1 per cent on iodine value.

Starch Solution.—Stir up 2 to 3 g. of potato starch with 100 cc. of 1-per-cent salicylic-acid solution, and boil the mixture till starch is practically dissolved, then dilute to 1 liter,[1] or as per Lord.[2]

[1] *Lead Peroxide.*—If sample contains an appreciable amount of nitrite (nitrate has no effect on method), leach out water-soluble matter as below, dry residue and determine PbO_2 as above, calculating to basis of original sample.

[2] "Notes on Metallurgical Analysis," p. 103 (1903).

DETERMINATION OF ZINC.

Zinc. 7. If in appreciable amount, evaporate off the alcohol from the filtrate from total lead, make alkaline with NH_4OH, then acid with HCl, add 3 cc. more of concentrated HCl, dilute to about 250 cc. with water, heat nearly to boiling and titrate with standard $K_2Fe(CN)_6$ solution as described by Low.[1] Report as ZnO (includes cadium). Iron, copper or other interfering substances should first be removed as described by Low.

WATER-SOLUBLE.

Water-Soluble. 8. Digest 10 g. of sample with 200 cc. of hot water on steam bath for 1 hour; filter on an 11-cm. S. & S. blue-ribbon paper and wash with hot water till no residue is left on evaporating a few drops of the washings. Evaporate filtrate to dryness on steam bath in a weighed dish, dry 30 minutes at 105° C., cool and weigh. Take up with water and if alkaline, titrate with tenth normal acid and methyl orange; calculate to Na_2CO_3.

Another lot of water-soluble matter is tested for nitrates, nitrites, carbonates, sulfates, sodium and lead.

TOTAL SILICA.

Total Silica. 9. Digest 5 g. of the sample in a covered casserole with 5 cc. of HCl and 15 cc. of HNO_3 (1 : 1). Evaporate to dryness to dehydrate. Cool, treat with hot water and HNO_3, boil, filter, wash with hot acid ammonium-acetate solution, then dilute HCl and finally hot water. Ignite and weigh as SiO_2. The residue may be treated with H_2SO_4 and HF in cases of doubt as to purity.

CARBON DIOXIDE.

Carbon Dioxide. 10. Determine carbon dioxide by the evolution method, using dilute HCl and stannous chloride.

DETERMINATION OF SOLUBLE SULFATE.

Soluble Sulfate. 11. *Sulfates other than Barium Sulfate.*—Treat 0.5 g. of the sample with 5 cc. of water, 3 g. of NH_4Cl and 5 cc. of HCl saturated with bromine; digest (covered) on steam bath about 15 minutes, add 25 cc. of water, neutralize with dry Na_2CO_3, and add about 2 g. more, boil 10 to 15 minutes; let settle, dilute with hot water, filter and wash with hot water; redissolve in

[1] Low, "Technical Methods of Ore Analysis."

HCl, reprecipitate as above and wash thoroughly with hot water; acidify united filtrates with HCl, adding a slight excess; boil and add slight excess of 10-per-cent $BaCl_2$ solution. Let stand on steam bath 1 hour, filter, wash with hot water, ignite, and weigh $BaSO_4$. Calculate to SO_3 (includes SO_3 formed from SO_2).

Or, dissolve 0.5 g. of the sample in 25 cc. of water, 10 cc. of NH_4OH (sp. gr. 0.90) and HCl in slight excess; dilute to about 150 cc. with water and add a piece of aluminum foil which should about cover the bottom of the beaker—this held on bottom by means of a stirring rod. Heat gently till all lead is precipitated, decant through a filter, pressing the lead sponge with a flattened rod, and washing with hot water. Add to the filtrate a little pure bromine water, boil until bromine is expelled, add 15 cc. of 10-per-cent $BaCl_2$ solution, let stand on steam bath one hour, filter, wash with hot water, ignite and weigh as $BaSO_4$ (any $SrSO_4$ present is not decomposed in this method).[1]

IRON OXIDE.

Iron Oxide.

12. Determine iron oxide by Schaeffer's[2] modification of Thomson's colorimetric method; or, in a large beaker, treat 20 g. of the sample with 20 cc. of water, 20 cc. of HNO_3 (sp. gr. 1.4) and 3 cc. of formaldehyde solution. Warm till all PbO_2 is dissolved, dilute with water, warm, filter off insoluble anp wash with hot water. Ignite filter and insoluble, evaporate with H_2SO_4 and HF. To filtrate from insoluble add 14 cc. of H_2SO_4 (1 : 1), filter off $PbSO_4$, and wash. Dissolve residue from HF and H_2SO_4 in H_2SO_4 and add to filtrate from $PbSO_4$; dilute to 500 cc. and determine iron colorimetrically in an aliquot, using same amounts of HNO_3, H_2SO_4 and formaldehyde in comparison solution.[3] Calculate to Fe_2O_3.

[1] The solubility of $BaSO_4$ is increased by the presence of aluminum chloride. See J. W. Mellor, "A Treatise on Quantitative Inorganic Analysis," p. 615.

[2] *Journal of Industrial and Engineering Chemistry*, Vol. 4, p. 659 (1912).

[3] Lunge-Berl, "Chemisch-technische Untersuchungs-Methoden," Bd. 2, S. 95, 6th Ed.

STANDARD METHODS OF ROUTINE ANALYSIS OF YELLOW, ORANGE, RED, AND BROWN PIGMENTS CONTAINING IRON AND MANGANESE[1]

Serial Designation: D 50 - 27

These methods are issued under the fixed designation D 50; the fina number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1917; ADOPTED, 1918; REVISED, 1927.

These methods were approved April 30, 1923, as "Tentative American Standard" by the American Engineering Standards Committee.

GENERAL METHODS.

SPECIFIC GRAVITY.

Specific Gravity.

1. True specific gravity shall be determined in accordance with the Standard Methods of Test for Specific Gravity of Pigments (Serial Designation: D 153) of the American Society for Testing Materials.[2]

TINTING STRENGTH.[3]

Tinting Strength

2. The colors should always be compared with a carefully selected standard. Weigh out accurately from 0.02 to 0.05 g. of color, place on a large glass plate, add 12 drops of bleached linseed oil,[4] and rub up with a flat-bottom glass pestle or muller; then add 1 g. of pure ZnO, and grind with a circular motion 50 times; gather up with a sharp-edge spatula and grind out twice more in like manner, giving the pestle a uniform pressure. Next weigh out a similar amount of the standard, and treat in exactly the same manner as described above. Transfer portions of each paste to a microscope slide, quite close together, and then draw a palette knife across both samples so as to make them meet in a line (differences in tint are then easily seen). Compare the tints as shown on both sides of the glass. The amount of pigment

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

[2] See p. 277.

[3] Holley, "Analysis of Paint and Varnish Products," 1912, p. 126.

[4] The use of "oxidized" oil should be avoided (oil that has become "fatty" by standing around in a can or bottle). Poppy-oil could be used but it is quite as necessary to have it reasonably fresh as when linseed oil is used. In either case "stringy" pastes are not reliable.

used will vary with the tinting strength, but generally it suffices to take about 0.02 g. of the reds and about 0.05 g. of the ochers.

If it is desired to express quantitatively the tinting strength of the pigment, consider the standard pigment as having a tinting strength of 100, and then vary the amount of the standard pigment until the tints are matched.

For example, let the amount of standard pigment for 100 strength test be 0.02 g. ($= A$). Vary this amount in units of 5 per cent, thus 0.019 or 0.021. Let the amount taken to match a given strength $= B$, then

$$\frac{B}{A} \times 100$$

equals the tinting strength of the sample to be tested.

If the tone varies it may be difficult to make this measurement.

ADDED COLORING MATTER.

Added Coloring Matter.

3. Test the pigment successively with hot water, 95-per-cent ethyl alcohol, alcoholic NaOH or KOH and acetic acid. Chloroform, NaOH, H_2SO_4, HCl-stannous chloride, and other reagents may be tried.[1] The presence of an organic color may often be noted by the characteristic odor given off on ignition.

METHODS OF ANALYSIS OF INDIAN REDS, RED OXIDES (PRINCE'S METALLIC, TUSCAN RED).

LOSS AT 100° C.

Loss at 100° C.

4. Heat 2 g. in a steam-jacketed oven at atmospheric pressure for three hours, or to constant weight.

LOSS ON IGNITION.

Loss on Ignition.

5. Ignite a portion in a covered porcelain crucible to constant weight. This may include combined water, CO_2, organic matter, and some SO_3 if much $CaSO_4$ is present. CO_2 may be determined on a separate portion of the sample if desired.[2]

FREE ACID OR ALKALI.

Free Acid or Alkali.

6. Boil 10 g. of sample with 100 cc. of water; filter and wash. Test filtrate with litmus paper; if acid, titrate with standard alkali and methyl orange and calculate to the equivalent of H_2SO_4. If alkaline, titrate with acid and calculate to the equivalent of Na_2O. Test filtrate for alkali salts and alkaline earths.

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[1] For details consult Zerr, "Tests for Coal-Tar Colors in Aniline Lakes," (English translation by C. Mayer); Schultz and Julius, "A Systematic Survey of the Organic Coloring Matters;" Hall, "The Chemistry of Paints and Paint Vehicles;" and Mulliken, "Identification of Pure Organic Compounds." *Commercial Dyestuffs*, Vol. III.

[2] It is inadvisable to use platinum unless it is known that attacking substances are absent.

ADDED COLORING MATTER.

7. Test as under "General Methods." **Added Coloring Matter.**

INSOLUBLE, IRON OXIDE, ETC.

8. Digest 2.5 g. of the sample (previously roasted at a low temperature if much organic matter is present; if very low in carbonaceous matter a little $KClO_3$ or $NaClO_3$ may be used in effecting solution) with 25 cc. of HCl (adding a little HNO_3 or chlorate, if not already added), wash off cover, and evaporate to dryness. Take up with HCl and water, filter, wash with dilute HCl and cold water. Make the filtrate up to 500 cc., mix, and examine as below.[1] Ignite the residue and weigh as "insoluble matter";[2] if this contains $BaSO_4$ it may be determined by fusing with six times its weight of Na_2CO_3, cooling, digesting with hot water, filtering, and washing the residue with hot water until free of sulfate. **Insoluble, Iron Oxide, etc.**

Remove filtrate and place beaker used for the digestion underneath the funnel, pierce the filter with the glass rod, and wash the residue with a little water into the beaker; then pour hot dilute HCl (1 : 1) over paper, and finally wash with hot water. If necessary add more HCl to the beaker to dissolve the $BaCO_3$; heat to boiling, add dilute H_2SO_4 in slight excess, let stand about one hour on steam bath; filter, wash, dry, ignite, and weigh $BaSO_4$. (This subtracted from total insoluble will give "insoluble silicious matter," if it is desired to so report.) If it is desirable to analyze the insoluble silicious matter, this can be done by the usual methods for silicate analysis, but the results should be reported as a separate analysis.

For the determination of iron place 100 cc. of the first filtrate in a flask, add about 3 g. of granulated zinc, put a funnel into the neck of the flask, heat when the action slackens; if basic salts separate out add a few drops of HCl. When the reduction is complete, add 30 cc. of H_2SO_4 (1 : 2), and as soon as the residual zinc is dissolved, wash down the funnel inside and out and the neck of the flask with a fine jet of water, filling the flask (1000 cc.) about two-thirds full, cool in water, add 10 cc. of "titrating solution" (made by dissolving 160 g. of manganese sulfate in water, diluting to 1750 cc., adding 330 cc. of H_3PO_4, sp. gr. 1.72, and 320 cc. of concentrated H_2SO_4), and titrate with

[1] For more exact work this filtrate should be evaporated to dryness and SiO_2 removed.

[2] If the insoluble contains appreciable amounts of Fe it will be necessary to fuse it with Na_2CO_3 or $K_2S_2O_7$ to determine total Fe in samples.

$KMnO_4$ (5.659 g. per liter) that has been standardized against Bureau of Standards sodium oxalate. Run a blank on the zinc, correct for same and calculate total iron as Fe_2O_3. Instead of adding the zinc to the solution, the reduction may be effected in a zinc reductor.[1]

The Fe_2O_3 may also be determined by the $K_2Cr_2O_7$ method.[2]

LIME.

Lime.

9. Dilute an aliquot of 100 cc. of the original solution to about 200 cc., add 10 cc. of HCl, make alkaline with HN_4OH, add 2 or 3 cc. of bromine water, and boil till excess of NH_3 is expelled. Let settle, wash by decantation, redissolve in HCl, and reprecipitate with NH_4OH and bromine water. (Precipitate equals $Fe_2O_3 \cdot Al_2O_3 \cdot TiO_2 \cdot P_2O_5 \cdot MnO_2$.) This precipitate may be ignited and weighed if desired.

To the combined filtrates add a few drops of NH_4OH, heat to boiling, and add an excess of saturated ammonium-oxalate solution; continue the boiling until the precipitate becomes granular, let stand about 30 minutes, filter, and wash with hot water till free of ammonium oxalate;[3] place beaker in which precipitation was made under the funnel, pierce apex of filter with stirring rod and wash precipitate into beaker with hot water, pour warm dilute H_2SO_4 (1 : 4) through paper and wash a few times; add about 30 cc. of H_2SO_4 (1 : 4), dilute to about 250 cc., heat to 90° C. and titrate at once with standard $KMnO_4$ solution (solution should not be below 60° C. when end-point is reached). Calculate to CaO. (The Fe value of $KMnO_4 \times 0.502 = CaO$.) The calcium-oxalate precipitate may be ignited to constant weight as CaO. If desired, magnesia may be determined as $Mg_2P_2O_7$ in the usual manner in the filtrate from the calcium oxalate.[4]

SOLUBLE SULFATES.

Soluble Sulfates.

10. Treat 1 g.[5] of the pigment (roasted gently if much organic matter is present) with 30 cc. of HCl, boil 10 minutes, add about 50 cc. of water, boil, filter, and wash with hot water. Heat the

[1] Lord and Demorest, "Metallurgical Analysis," 1913, pp. 28–29.

[2] *Ibid.*, pp. 21–26.

[3] For more exact work this precipitate should be dissolved in HCl and the calcium oxalate reprecipitated as above.

[4] If desired, a direct determination of Al_2O_3 may be made on an aliquot of the solution or on the HCl solution of the NH_4OH precipitate by Peters' phosphate method (this will include titanic acid) as described by Blair, "The Chemical Analysis of Iron," and Philips, "Methods of Iron Analysis Used in the Pittsburgh District."

[5] If low in soluble sulfates use a larger portion of sample.

solution to boiling, add NH_4OH, filter and wash a few times with hot water; dissolve precipitate in hot dilute HCl and reprecipitate with NH_4OH, wash well with hot water. Render united filtrates just distinctly acid with HCl, boil, add by drops with stirring excess of 10-per-cent $BaCl_2$ solution, boil about 10 minutes, filter on a Gooch crucible, wash with hot water, ignite and weigh as $BaSO_4$. Calculate to SO_3 or $CaSO_4$.

TOTAL SULFUR OTHER THAN THAT PRESENT AS $BaSO_4$.

11. Treat 5 g. of the sample in a covered porcelain dish with 50 cc. of aqua regia (1 HNO_3 : 9 HCl) and evaporate to dryness on steam bath. Add 20 cc. of concentrated HCl and about 250 cc. of water, make double NH_4OH precipitation; determine $BaSO_4$ as given under "Soluble Sulfates." **Total Sulfur.**

METHODS OF ANALYSIS OF OCHERS.

12. Loss at 100° C., loss on ignition, insoluble matter, total or soluble iron, alumina, lime and sulfur may be determined as outlined under the "Methods for Analysis of Indian Reds, etc.," using 1 g. or an aliquot corresponding to this weight.

TINTING STRENGTH.

13. Test as under "General Methods." Tests should be made for solubility in water, and reaction to litmus paper. **Tinting Strength.**

LEAD CHROMATE.

14. If present, the lead is removed in the above scheme by nearly neutralizing the filtrate from the insoluble matter with NH_4OH, cooling, and passing in H_2S, to precipitate PbS. Filter, wash with water containing H_2S, dissolve PbS in hot dilute HNO_3, add 10 cc. of concentrated H_2SO_4, evaporate till SO_3 is evolved, cool, dilute to 200 cc., let stand a few hours, filter on a Gooch crucible, wash with 1-per-cent H_2SO_4, ignite, and weigh $PbSO_4$. Calculate to PbO or Pb. Heat the filtrate from the PbS to expel H_2S, oxidize with a little HNO_3, and make up to volume if working on more than 1 g. **Lead Chromate.**

The iron is best determined in an aliquot by the $K_2Cr_2O_7$ method. Another aliquot is treated with NH_4OH, the precipitate containing $Al_2O_3 \cdot Fe_2O_3 \cdot Cr_2O_3 \cdot P_2O_5 \cdot TiO_2$. Lime and MgO may be determined in filtrate.

The NH_4OH precipitate is dissolved in hot dilute HCl, washing paper with hot water, *cooled*, oxidized with Na_2O_2,

boiled to expel H_2O_2, cooled, cover glass washed off, diluted to about 150 cc., and acidified with H_2SO_4. Add a measured excess of ferrous ammonium-sulfate solution—$(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$, 12.4 g.; concentrated H_2SO_4, 50 cc.; and water to make 1 liter, and titrate back with standard $K_2Cr_2O_7$ solution, using $K_3Fe(CN)_6$ solution as an outside indicator. The $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$ solution is titrated with the $K_2Cr_2O_7$ solution to determine its value in terms of the latter. The Fe value of the $K_2Cr_2O_7$ solution $\times 0.597 = CrO_3$.

Or, moisten 1 g. of the pigment with water, add 5 cc. of concentrated HCl, boil a few minutes, *cool*, add Na_2O_2 in excess, boil to expel H_2O_2, cool, wash off cover glass, dilute, acidify with H_2SO_4, and titrate CrO_3 as above.

VENETIAN RED.

Venetian Red.

15. Analyze as given under "Methods for Analysis of Indian Reds, etc." Insoluble matter may be treated with HF and H_2SO_4 to determine SiO_2 by loss if desired.

METHODS OF ANALYSIS OF SIENNAS AND UMBERS.

16. After gently roasting to destroy organic matter, test as given under "Methods for Analysis of Indian Reds, etc."

MANGANESE.

Manganese.

17. Manganese is determined by the bismuthate method.[1] Ignite gently (to destroy organic matter) 1 g. of the sample in a platinum dish, cool, add 10 cc. of water, 4 cc. of concentrated H_2SO_4 and about 20 cc. of HF (if necessary, add a little sulfurous acid). Evaporate until the H_2SO_4 fumes freely, cool and dissolve in 25 cc. of HNO_3 (1 part concentrated HNO_3 to 3 parts water). If no appreciable residue remains, transfer to a 100-cc. volumetric flask, using 25 cc. of HNO_3 (1 : 3) to rinse the dish, dilute to the mark with water, mix thoroughly. If there is an appreciable residue, filter on a small filter, wash with water, ignite residue in a platinum crucible, and fuse with a little sodium or potassium pyrosulfate. Dissolve in water, with the addition of a little HNO_3, add to the main filtrate, evaporate nearly to dryness, take up in HNO_3 (1 : 3) and transfer to the flask as before. Pipette an aliquot of 10 cc. into a

[1] Blair, "The Chemical Analysis of Iron."

200-cc. Erlenmeyer flask, add 30 cc. of water and 10 cc. of concentrated HNO_3, sp. gr. 1.4; add about 0.5 g. of sodium bismuthate, heat for a few minutes, or until the pink color has disappeared with or without the precipitation of MnO_2. Add a few small crystals of sodium or potassium nitrite to dissolve the MnO_2 and boil the solution several minutes to expel nitrous fumes (a little Na_2CO_3 will aid this). Add water to bring the volume up to 50 cc. and cool to about 15° C.; add about 0.5 g. of bismuthate and shake the flask well. Add 50 cc. of water containing 30 cc. of concentrated HNO_3 to the liter, filter by suction through an asbestos felt into a 300-cc. Erlenmeyer flask and wash with 50 to 100 cc. of the same acid. Run in a measured volume of standard ferrous ammonium-sulfate solution and titrate to a faint pink color with standard $KMnO_4$ solution. The number of cubic centimeters of the $KMnO_4$ solution obtained, subtracted from the number corresponding to the volume of ferrous solution used, will give the volume of $KMnO_4$ equivalent to the manganese in the sample, which, multiplied by the value of the $KMnO_4$ in Mn, gives the weight of manganese in the portion of sample used.

Standard $KMnO_4$ Solution.—The solution of $KMnO_4$ is composed of 1 g. dissolved in a liter of water. The Fe value of this solution $\times$ 0.197 = Mn. This solution may be standardized against Bureau of Standards sodium oxalate (using about 0.05 to 0.1 g.).[1] Weight of $Na_2C_2O_4 \times 0.164$ = Mn. Twelve grams of $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$, 25 cc. of concentrated H_2SO_4, and 25 cc. of H_3PO_4, sp. gr. about 1.7, are made up to 1 liter with water. The value of this solution should be determined against the $KMnO_4$ each day as follows:

Measure into a 200-cc. Erlenmeyer flask 50 cc. of HNO_3 (1 : 3), cool, add a little bismuthate, dilute with 50 cc. of 3-per-cent HNO_3, filter by suction through an asbestos felt into a 300-cc. Erlenmeyer flask, and wash with 50-cc. of 3-per-cent HNO_3. Run in 25 cc. of the ferrous solution and titrate with $KMnO_4$ solution. Instead of titrating the permanganic acid formed by the bismuthate with the ferrous solution and then titrating back with $KMnO_4$, a direct titration with standard sodium arsenite solution may be made.[2]

[1] W. Blum, "Original Communications," Eighth International Congress of Applied Chemistry, Vol. I, pp. 61–85.

[2] Lord and Demorest, "Metallurgical Analysis," 1913, p. 82

STANDARD METHODS OF ROUTINE ANALYSIS OF YELLOW AND ORANGE PIGMENTS CONTAINING CHROMIUM COMPOUNDS, BLUE PIGMENTS AND CHROME GREEN[1]

Serial Designation: D 126 – 27

These methods are issued under the fixed designation D 126; the final number indicates the year of original adoption as standard, or in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED IN AMENDED FORM, 1923; REVISED, 1927

GENERAL METHODS.

SPECIFIC GRAVITY.

Specific Gravity.

1. True specific gravity shall be determined in accordance with the Standard Methods of Test for Specific Gravity of Pigments (Serial Designation: D 153) of the American Society for Testing Materials.[2]

TINTING STRENGTH.[3]

Tinting Strength.

2. The colors should always be compared with a carefully selected standard. Weigh out accurately a portion of the color (0.02 g. of yellow; 0.01 g. of green or blue), place on a large glass plate, add 24 drops of bleached linseed oil[4] (36 drops with blues), and mix with a clean steel spatula until the mass appears to be homogeneous; add pure ZnO (2 g. for yellows or greens, 3 g. for blues), and grind with a circular motion 50 times, using a flat-bottomed glass pestle or muller; gather up with a sharp-edged spatula and grind out twice more in like manner, giving the pestle a uniform pressure. Next weigh out a similar amount of the standard, and treat in exactly the same manner as described above. Transfer portions of each paste to a microscope

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

[2] See p. 277.

[3] Holley, "Analysis of Paint and Varnish Products," 1912, p. 126.

[4] The use of "oxidized" oil should be avoided (oil that has become "fatty" by standing in a can or bottle). Poppy oil could be used but it is quite as necessary to have it reasonably fresh as when linseed oil is used. In either case "stringy" pastes are not reliable.

slide, quite close together, and then draw a palette knife across both samples so as to make them meet in a line (differences in tint are then easily seen). Compare the tints as shown on both sides of the glass. The amount of pigment used will vary with the tinting strength, but the amounts indicated will generally suffice.

If it is desired to express quantitatively the tinting strength of the pigment, consider the standard pigment as having a tinting strength of 100, and then vary the amount of the standard pigment until the tints are matched.

For example, let the amount of standard pigment for 100 strength test be 0.05 g. ($= A$). Vary this amount in units of 5 per cent, thus 0.0475 or 0.0525. Let the amount required to match a given strength equal B, then

$$\frac{B}{A} \times 100$$

equals the tinting strength of the sample to be tested.

If the tone varies it may be difficult to make this measurement.

ADDED COLORING MATTER.

3. Test the pigment successively with hot water, 95-per-cent ethyl alcohol, and chloroform. The solutions should remain colorless. Other reagents may be tried.[1] **Added Coloring Matter.**

METHODS OF ANALYSIS OF YELLOW AND ORANGE PIGMENTS.

CHROME YELLOWS, AMERICAN VERMILION, BASIC LEAD CHROMATE.

A pure chrome yellow should contain only lead chromate and other insoluble lead compounds.

MOISTURE.

4. Heat 2 g. at 105° C. for two hours. The loss in weight is reported as moisture. **Moisture.**

INSOLUBLE MATTER.

5. Treat 1 g. with 25 cc. of concentrated HCl and boil for from 5 to 10 minutes in a covered beaker, adding about 6 drops of alcohol to the boiling liquid, one at a time. Dilute to 100 cc. with hot water and boil for from 5 to 10 minutes (the solution should be complete). Filter the hot solution (if insoluble matter is present) and wash with boiling water till washings are free from lead and chlorine. Ignite the insoluble matter, weigh, and examine for SiO_2, $BaSO_4$, and Al_2O_3. **Insoluble Matter.**

[1] For details consult Zerr, "Tests for Coal-Tar Colors in Aniline Lakes" (English translation by C. Mayer); Schultz and Julius, "A Systematic Survey of the Organic Coloring Matters;" Hall, "The Chemistry of Paints and Paint Vehicles;" and Mulliken, "Identification of Pure Organic Compounds," *Commercial Dyestuffs*, Vol. III.

TOTAL LEAD.

Total Lead. 6. Add NH_4OH to the filtrate from the insoluble matter (or the original solution) until a faint precipitate begins to form, then add 5 cc. of concentrated HCl (sp. gr. 1.19), dilute to 500 cc., and pass into the clear solution a rapid current of H_2S until all of the lead is precipitated as PbS. Let the precipitate settle, filter, wash with water containing some H_2S. Boil the filter and precipitate with dilute HNO_3 until all of the lead has dissolved, filter, and wash thoroughly with hot water. To the filtrate, add 10 cc. of H_2SO_4 (1 : 1), evaporate until copious fumes of SO_3 are evolved, cool, add about 75 cc. of H_2O and then 75 cc. of 95-per-cent ethyl alcohol. Let stand about one hour, filter on a Gooch crucible, wash with dilute alcohol, dry, ignite, and weigh as $PbSO_4$. Save the alcoholic filtrate from the $PbSO_4$, evaporate nearly to dryness and add to the filtrate from the PbS.

CHROMIUM (IRON, ALUMINUM).

Chromium. 7. Heat the filtrate from the PbS to expel H_2S and, if iron is present, add a few drops of HNO_3 and boil about two minutes. Render the solution just alkaline with NH_4OH, boil a few minutes, filter, and wash with hot 2-per-cent NH_4Cl solution. (If the sample contains an appreciable amount of zinc, a double precipitation should be made.) In the absence of iron and aluminum this precipitate may be ignited and weighed as Cr_2O_3. If iron and aluminum are present, dissolve the NH_4OH precipitate with hot dilute HCl, washing the paper with hot water; evaporate to about 100 cc., *cool*, add NH_4OH until alkaline, and then add Na_2O_2 (containing at least 90 per cent of Na_2O_2) in small portions to the cooled solution (10 to 12° C.) until oxidation is complete, keeping the beaker covered. Digest until all of the chromium and aluminum has been dissolved, adding more Na_2O_2 if necessary. Filter off the $Fe(OH)_3$, wash thoroughly with hot water, ignite and weigh as Fe_2O_3; or, dissolve the precipitate in HCl and determine the Fe content volumetrically. Make up the filtrate from the $Fe(OH)_3$ to 250 cc. in a graduated flask, and mix. Render an aliquot portion acid with H_2SO_4, boil to expel any free oxygen, cool, add an excess of standard $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$ solution and titrate back with 0.1 *N* $K_2Cr_2O_7$ solution, using $K_3Fe(CN)_6$ as outside indicator. (The CrO_3 may also be determined by acidifying the aliquot portion with acetic acid, precipitating as $PbCrO_4$ or $BaCrO_4$, and finally weighing on a Gooch crucible.) To determine Al_2O_3, make an aliquot portion of the filtrate from the $Fe(OH)_3$ acid with HCl, and then just distinctly alkaline with NH_4OH, heat to boiling, let settle, filter, wash with hot 2-per-cent NH_4Cl solution, ignite and weigh as Al_2O_3. If

iron and aluminum are not to be determined or are present in negligible amounts, the first NH_4OH precipitate may be dissolved in dilute HCl, oxidized with Na_2O_2, acidified with H_2SO_4, boiled, and CrO_3 determined volumetrically. The CrO_3 in the absence of other oxidizing substances, may be determined on 1 g. of the pigment by Schwartz' method (Fresenius Quantitative Chemical Analysis, Ed. 6, Vol. 1, p. 424) or by the persulfate method,[1] as follows:

For c. p. chrome yellows use an 0.25-g. sample, for c. p. chrome greens use an 0.5-g. sample, and for commercial chrome greens use a 1-g. sample. Weigh the sample, transfer to a 600-cc. Pyrex beaker, add 25 cc. of concentrated H_2SO_4, and fume lightly on a hot plate for 3 or 4 minutes. Excessive fuming or *cooking* over a hot flame is neither necessary nor desirable, as it may produce the difficultly soluble anhydrous chromium sulfate. The Prussian blue color is destroyed in a few minutes. Cool, cautiously dilute with water to 300 cc., stir, and heat to boiling. To the boiling solution add a small amount of permanganate (0.5 to 1 cc. of 0.1 *N* $KMnO_4$) to insure the presence of some manganese. Then add to the boiling solution 10 cc. of $AgNO_3$ solution (2.5 g. in enough water to make 1 liter) and 20 cc. (adding this slowly) of ammonium persulfate solution (4 g. of ammonium persulfate of full strength dissolved in 20 cc. of water). The hot solution should show the usual permanganate color. If this color does not develop, or if it disappears, add more persulfate. When the permanganate color is permanent, continue the boiling (best done on a hot plate) for 10 to 15 minutes to destroy excess persulfate. Then add 5 cc. of dilute HCl (1:3) and boil for 5 to 8 minutes to reduce the oxidized manganese. The color now should be the usual chromate yellow. Cool to about 20° C. and titrate either electrometrically with ferrous sulfate,[2] or add a measured excess of ferrous sulfate solution (beyond the deep grass-green color) and titrate back with 0.1 *N* permanganate. The first faint permanent darkening of the green color is taken as the end point.

Correction for a blank (about 0.2 cc. of $KMnO_4$) due to color, etc., should be made.

NOTES

In commercial chrome greens containing silicates and barium sulfate, filtration to remove the "acid insoluble matter" after fuming the sample with acid and diluting is advisable though not necessary. The end point of the subsequent titration of

[1] E. F. Hickson, *Circular No. 294*, Scientific Section, Am. Paint and Varnish Manufacturers' Assn., November, 1928.

[2] Kelly, *Journal of Industrial and Engineering Chemistry*, Vol. 13, p. 1053 (1921).

the clear solution is easier to see, and there is less chance for "bumping" during boiling if these are removed.

In the case of c. p. greens and yellows, the solution, after fuming with sulfuric acid and diluting, could be filtered and the precipated lead sulfate weighed directly for total lead. This would likewise give a clear filtrate for the chromium titration.

The important precautions are to avoid overheating with sulfuric acid, and to make sure that the persulfate (solid material) has not deteriorated. It should contain over 90 per cent of the reagent.

ZINC, CALCIUM, AND MAGNESIUM.

Zinc, Calcium, Magnesium.

8. Precipitate any zinc in the filtrate from the first NH_4OH precipitate with H_2S, filter, wash with dilute $(NH_4)_2S$, dissolve the zinc sulfide in dilute HCl, and determine the Zn content volumetrically by $K_4Fe(CN)_6$ method. In the filtrate from the zinc sulfide, determine calcium by the oxalate method and magnesium as $Mg_2P_2O_7$.

SULFURIC ANHYDRIDE.

Sulfuric Anhydride.

9. Heat 1 g. of the pigment with 10 cc. of concentrated HCl until free chlorine is expelled, add about 300 cc. of water and boil; filter off any insoluble matter and wash thoroughly with hot water, heat to boiling, and precipitate with $BaCl_2$ solution in the usual manner. Keep the solution hot while filtering off the $BaSO_4$ and wash with hot water until the washings show no lead or chlorine.

CARBON DIOXIDE.

Carbon Dioxide.

10. Determine carbon dioxide by the evolution method, using dilute HNO_3, free from oxides of nitrogen.

WATER-SOLUBLE MATTER.

Water-Soluble Matter.

11. Weigh 2.5 g. of the pigment and transfer to a graduated 250-cc. flask, add 100 cc. of water, and boil for 5 minutes. Dilute with water, let stand until at room temperature, make up to the mark with water, mix, and let settle. Filter through dry paper and discard the first 25 cc. Transfer 100 cc. of the clear filtrate to a weighed dish, evaporate to dryness on a steam bath, dry in an oven at 105 to 110° C. to constant weight (30 minutes will usually suffice); cool and weigh.

TINTING STRENGTH AND ADDED COLORING MATTER

Coloring Matter.

12. Make the test for added coloring matter and tinting strength in accordance with Sections 2 and 3.

CALCULATIONS.

Calculations.

13. Calculate CrO_3 to $PbCrO_4$, and SO_3 to $PbSO_4$ if calcium is absent. If CO_2 is present and calcium and magnesium are absent,

calculate to $(PbCO_3)_2 \cdot Pb(OH)_2$. Report any residual Pb as PbO. If calcium is present, calculate to $CaCO_3$ if CO_2 is also present. If calcium and SO_3 are present and CO_2 is absent, calculate to $CaSO_4$. If calcium, CO_2, and SO_3 are present, calculate to $CaCO_3$; any residual calcium is then calculated to $CaSO_4$. Report zinc as ZnO.

METHODS OF ANALYSIS OF BLUE PIGMENTS.

IRON CYANIDE BLUES. (PRUSSIAN BLUE, CHINESE BLUE, ANTWERP BLUE, MILORI BLUE, BRONZE BLUE, STEEL BLUE.)

The analysis of these blues, as is generally the case with pigments, does not necessarily give results which can be used to grade samples, the strength and color tests being most important.

MOISTURE.

Moisture.

14. Heat 2 g. of the pigment at 105° C. for two hours. The loss in weight is reported as moisture. A "dry" Prussian blue should contain less than 7 per cent of moisture.

INSOLUBLE MATTER.

Insoluble Matter.

15. Ignite 1 g. of the pigment in a porcelain dish at a low temperature, just high enough to decompose the last trace of blue, but not high enough to render the iron difficultly soluble in HCl. Cool, add 15 cc. of HCl and a few drops of bromine, cover with a watch glass, and digest on the steam bath; wash off cover, evaporate to a syrup, add water, boil, filter, wash with hot water, ignite the residue and weigh. Examine the insoluble residue for silica, barium sulfate, and alumina. A pure Prussian blue should show no insoluble residue.

IRON AND ALUMINUM.

Iron and Aluminum.

16. Determine iron and aluminum in the filtrate from the insoluble matter by precipitation with NH_4OH in the usual manner. A double precipitation is desirable. Ignite and weigh $Fe_2O_3+Al_2O_3$, deduct Fe_2O_3 (calculated from total Fe), and calculate Al_2O_3 to Al.

CALCIUM.

Calcium.

17. Determine calcium in the filtrate from the $Fe_2O_3+Al_2O_3$ by precipitation with ammonium oxalate; titrate with $KMnO_4$, or, ignite and weigh as CaO. Acidify the filtrate from the calcium oxalate with HCl and dilute to a definite volume and mix.

SULFURIC ACID.

Sulfuric Acid.

18. Determine sulfuric acid in an aliquot of the above solution as $BaSO_4$ in the usual manner.

ALKALI METAL AND ALKALINE SALTS.

Alkali.

19. Evaporate an aliquot of the above solution with sulfuric acid, ignite (treating with solid ammonium carbonate), and weigh. Determine whether the alkali metal is sodium or potassium and subtract the alkali metal corresponding to the sulfate (SO_4) found. The remainder is alkali combined with the blue and is reported as Na or K.

TOTAL IRON.

Total Iron.

20. Decompose and dissolve 1 g. of the pigment as under "Insoluble Matter," reduce, and determine the total iron with $KMnO_4$ or $K_2Cr_2O_7$. There should be not less than 30 per cent, calculated on the dry pigment.

TOTAL NITROGEN.

Total Nitrogen.

21. Determine the total nitrogen on a 1-g. sample of the pigment by the Kjeldahl-Gunning Method, digesting for at least $2\frac{1}{2}$ hours. The sulfuric acid should not blacken, which would indicate organic adulteration.

WATER-SOLUBLE MATTER.

Water-Soluble Matter.

22. Weigh 2.5 g. of the pigment into a graduated 250-cc. flask, add 100 cc. of water, and boil for five minutes. Dilute with water, let stand until at room temperature, make up to mark, mix and let settle. Filter through dry paper and discard the first 25 cc. Transfer 100 cc. of the clear filtrate to a weighed dish, evaporate to dryness on a steam-bath, dry in an oven at 105° C. for 30 minutes, cool, and weigh.

TINTING STRENGTH AND ADDED COLORING MATTER

Coloring Matter.

23. Make the test for added coloring matter and tinting strength in accordance with Sections 2 and 3, omitting the treatment with hot water. Also test the pigment with dilute acid for ultramarine blue (evolution of hydrogen sulfide) and carbonates (evolution of carbon dioxide).

CALCULATIONS.

Calculations.

24. The percentage of Prussian blue may be obtained with sufficient accuracy for commercial purposes by multiplying the percentage of nitrogen by 4.4 or the percentage of iron (in the absence of other iron pigments) by 3.03.[1]

NOTE.—Some blues, *e. g.*, Chinese blue, may contain tin salts. Others may contain manganese or chromium compounds. The presence of these compounds should be determined by a qualitative examination at least.

[1] Parry and Coste, *The Analyst*, Vol. 21, pp. 225 to 230 (1896).

METHODS OF ANALYSIS OF ULTRAMARINE BLUE.

An analysis is of little value for determining the quality of pure ultramarines, but is useful in the identification of foreign admixtures. Practical tests as to the stability and compatability of the pigment in mixtures with other pigments, coloring power, tint, fineness, fastness to light, etc., are more important than chemical analysis.

MOISTURE.

25. Heat 2 g. of the pigment at 105° C. for two hours, cool and weigh. The loss in weight is reported as moisture. **Moisture.**

SILICA.

26. Treat 1 g. of the pigment in a covered dish or casserole with 30 cc. of HCl (1:1), heat until decomposed, wash off and remove cover, and evaporate to dryness on the steam-bath. Moisten with concentrated HCl and again evaporate to dryness, add 1 to 2 cc. of concentrated HCl, let stand about 5 minutes, add hot water, filter and wash the insoluble matter with hot water. If great accuracy is desired, evaporate the filtrate to dryness, take up with HCl and water, filter on a second paper, wash, and add the residue to the main insoluble. Ignite the insoluble matter, cool and weigh. Determine SiO_2 by volatilization with H_2SO_4 and HF. Make a qualitative examination of any residue that may remain. **Silica.**

ALUMINA.

27. Render the filtrate from the silica faintly alkaline with NH_4OH, boil a few minutes, filter, wash with hot 2-per-cent NH_4Cl solution, ignite and weigh as $Al_2O_3(+Fe_2O_3)$. For more accurate work, dissolve the $Al(OH)_3$ precipitate in HCl and reprecipitate as above. **Alumina.**

SODIUM OXIDE.

28. Acidify the filtrate from the Al_2O_3 with H_2SO_4, evaporate to dryness, ignite (finally adding solid ammonium carbonate) and weigh as Na_2SO_4. Calculate to Na_2O. If calcium is present it should be precipitated with ammonium oxalate in the filtrate from the Al_2O_3, ignited and weighed as CaO, and the sodium determined in the filtrate from the calcium oxalate, as described. **Sodium Oxide.**

TOTAL SULFUR.

Total Sulfur.

29. Mix 1 g. of the ultramarine with 4 g. of Na_2CO_3 and 4 g. of Na_2O_2 in a nickel crucible, cover with about 1 g. of Na_2CO_3 and fuse, using an aluminum or asbestos shield to prevent the sulfur being taken up from the gas. Dissolve the fused mass in dilute HCl, filter and wash, if necessary (there should be no insoluble residue), precipitate with $BaCl_2$ and determine total sulfur by weighing as $BaSO_4$. Calculate to S.

SULFUR PRESENT AS SULFATE.

Sulfur as Sulfate.

30. Dissolve 1 g. of the pigment in dilute HCl, boil to expel H_2S, and filter if necessary; make the solution faintly alkaline, with NH_4OH and just distinctly acid with HCl, and treat with $BaCl_2$ in the usual manner. Calculate $BaSO_4$ to SO_3 and to S.

SULFUR PRESENT AS SULFIDE.

Sulfur as Sulfide.

31. Subtract the sulfur present as sulfate from the total sulfur.

METHODS OF ANALYSIS OF COBALT BLUE.[1]

This pigment is essentially a compound of the oxides of aluminum and cobalt. Certain shades of ultramarine blue are often sold under the name "cobalt blue."

MOISTURE.

Moisture.

32. Heat 2 g. of the pigment at 105° C. for 2 hours. The loss in weight is reported as moisture.

ALUMINA.

Alumina.

33. Fuse 1 g. of the pigment with 12 to 15 g of sodium or potassium pyrosulfate, cool, digest with water and HCl, filter, and wash the residue with hot water. Make the filtrate up to 250 cc. in a graduated flask and mix. Ignite the residue, cool, weigh, and examine for SiO_2 and $BaSO_4$. Dilute an aliquot portion of the filtrate to 200 cc., add 5 g. of NH_4Cl, heat to boiling, and add dilute NH_4OH till just distinctly alkaline (a few drops of 0.2-per-cent alcoholic solution of methyl red is recommended as indicator). Boil for one or two minutes, filter at once, dissolve the precipitate with HCl, and reprecipitate as before. Filter, wash thoroughly with hot 2-per-cent NH_4Cl (or NH_4NO_3) solution, ignite, and weigh as Al_2O_3.

[1] "Analysis of Paint and Varnish Products," C. D. Holley, p. 210 (1912).

CALCIUM AND MAGNESIUM.

34. Unite the filtrates from the Al_2O_3, saturate with hydrogen sulfide, filter, and determine calcium and magnesium in the filtrate in the usual manner. **Calcium and Magnesium.**

COBALT OXIDES.

35. Subtract the determined constituents from 100 and report the difference as cobalt oxides, unless a qualitative examination shows the presence of other substances in significant amounts. Should the pigment contain phosphoric acid (or arsenic acid) in more than negligible amounts, these must be removed before determining aluminum, calcium and magnesium.[1] **Cobalt Oxides.**

METHODS OF ANALYSIS OF SUBLIMED BLUE LEAD.[2]

TOTAL LEAD.

36. Digest 1 g. of the sample with 15 cc. of concentrated HNO_3 in a covered beaker. Boil the solution until the brown fumes of the oxides of nitrogen have disappeared. Add 6 cc. of concentrated H_2SO_4 and again boil until the heavy fumes of SO_3 are evolved. Allow the solution to cool, add 30 cc. of water and boil. Remove the beaker from the hot plate and allow the solution to stand for from 3 to 4 hours. Filter the solution, washing the precipitate by decantation 3 or 4 times and allowing the bulk of the precipitate to remain in the beaker. Place this beaker containing the residual lead sulfate under the funnel used for the filtration, wash the filter paper with 75 cc. of a mixture consisting of 95 cc. of NH_4OH (sp. gr. 0.90), 125 cc. of 80-per-cent acetic acid and 100 cc. of water. Follow this washing with 75 cc. of hot water. Boil until all $PbSO_4$ is dissolved. Dilute to 200 cc. with hot water, boil and titrate with standard ammonium molybdate solution, using a freshly prepared solution of one part of tannic acid in 300 parts of water as an outside indicator. Run a blank and correct for same. The ammonium molybdate solution contains 8.67 g. in one liter of water and is standardized against pure lead foil, pure PbO, or pure $PbSO_4$. **Total Lead.**

TOTAL SULFUR.

37. Treat 0.5 g. of the sample in a beaker with 10 cc. of water and a few cc. of bromine water. Boil gently until all the bromine has passed off. Dilute with water, add another portion of bromine water, boil, and continue the treatment until the sediment has become white **Total Sulfur.**

[1] See "Technical Methods of Chemical Analysis," Lunge-Keane, Vol. III, Part II, p. 978 (1914).

[2] "The Chemical Analysis of Lead and Its Compounds," Schaeffer and White, pp. 22 to 24.

in color. Add 8 cc. of HNO_3, evaporate the solution until the brown fumes of nitric acid have disappeared, dilute with water and add an excess of Na_2CO_3. Boil gently (covered) for from 10 to 15 minutes and let stand for 4 hours. Dilute with hot water, filter, and wash with hot water. Reject the residue. Acidify the filtrate (about 200 cc.) with HCl and add an excess of about 2 cc. of the acid. Boil and add a slight excess of 10-per-cent barium chloride solution. Let stand on a steam bath about 1 hour, filter, wash with hot water, ignite, and weigh as $BaSO_4$. Calculate the $BaSO_4$ to S.

LEAD SULFATE.

Lead Sulfate.

38. On a separate sample determine the sulfate directly with Na_2CO_3 as in the preceding paragraph, without any preliminary treatment with bromine water.

LEAD SULFITE.

Lead Sulfite.

39. Boil 1.5 g. of the sample with 3 g. of Na_2CO_3, let stand, filter, and wash thoroughly. To the filtrate add 3 cc. of bromine water, heat gently to oxidize the sulfite to sulfate, acidify with HCl, and precipitate with $BaCl_2$ solution. Filter, wash, ignite, and weigh as $BaSO_4$. Deduct the amount present as sulfate and calculate the remainder to lead sulfite.

LEAD SULFIDE.

Lead Sulfide.

40. Deduct the sulfur present as sulfate and sulfite from the total sulfur and report the difference as lead sulfide.

LEAD CARBONATE.

Lead Carbonate.

41. Determine any CO_2 present by the evolution method, removing any H_2S or SO_2 formed by means of $KMnO_4$ or CrO_3 solution. Calculate to $PbCO_3$.

LEAD OXIDE.

Lead Oxide.

42. Deduct the lead present as sulfate, sulfite, sulfide, and carbonate from the total lead and report the difference as lead oxide (PbO).

ZINC OXIDE.

Zinc Oxide.

43. Boil 1 g. of the sample with a solution of 4 g. of NH_4Cl in 30 cc. of water plus 6 cc. of concentrated HCl. Dilute to 200 cc. with hot water, add 2 cc. of a saturated sodium thiosulfate solution, and titrate with a standard solution of potassium ferrocyanide, using a 5-per-cent solution of a uranium nitrate as an outside indicator. Report as ZnO.

CARBON AND VOLATILE MATTER.

44. Ignite a weighed portion of the sample in a partially covered crucible at a low red heat for 2 hours, cool, and weigh. Report the loss in weight as carbon and volatile matter. **Carbon and Volatile Matter.**

METHODS OF ANALYSIS OF GREEN PIGMENTS.

(CHROME GREEN)

A pure chrome green should contain only Prussian blue and pure chrome yellow. A microscopic examination should be made to determine whether the green is a combined precipitation product, which is of the greater value, or one mixed after separate precipitation. A good green will show the presence of green and blue particles, while a poor green will show yellow and blue particles mixed with green.

MOISTURE.

45. Heat 2 g. of the pigment at 105° C. for two hours. The loss in weight is reported as moisture. **Moisture.**

INSOLUBLE MATTER.

46. Heat gently 1 g. of the pigment in a small porcelain dish until the blue color has been decomposed. The heating should be carried out very carefully so as not to render the iron difficultly soluble. (With some very pure chrome greens it may be advantageous to mix the sample with 2 to 5 times its weight of pure barium sulfate before igniting.) Let cool, transfer to a beaker, and determine insoluble matter as outlined in Section 5 for Yellow Pigments. **Insoluble Matter.**

LEAD.

47. Determine lead in the filtrate from the above as outlined in Section 6 for Yellow Pigments. **Lead.**

IRON, ALUMINA, AND CHROMIUM.

48. Determine iron, aluminum and chromium in the filtrate from the PbS as outlined in Section 7 for Yellow Pigments, making a double precipitation. **Iron, Alumina, Chromium.**

ZINC, CALCIUM, AND MAGNESIUM.

49. Determine zinc, calcium and magnesium in the filtrate from the iron, aluminum and chromium determination as outlined in Section 8 for Yellow Pigments. **Zinc, Calcium, Magnesium.**

CARBON DIOXIDE.

Carbon Dioxide

50. Determine carbon dioxide by the evolution method, using dilute HNO_3 (1 : 5).

SULFURIC ANHYDRIDE.

Sulfuric Anhydride.

51. Heat gently 1 g. of the pigment as in Sectn io46, cool, transfer to a beaker, add 30 cc. of concentrated HCl, cover, and heat on a steam-bath for about 30 minutes (in some cases, the iron compounds will go into solution more readily by letting the solution stand for some time at room temperature and then heating). Wash off cover, add 50 cc. of boiling water, boil for five minutes, filter, render the filtrate faintly alkaline with NH_4OH, then slightly acid with HCl, heat to boiling, and precipitate with $BaCl_2$ (15 cc. of 10-per-cent solution) in the usual manner, boiling about ten minutes. Filter, wash with hot water, ignite, and weigh the $BaSO_4$.

NITROGEN.

Nitrogen.

52. Determine nitrogen on a 1-g. portion of the pigment by the Kjeldahl-Gunning Method, digesting for at least $2\frac{1}{2}$ hours.

WATER-SOLUBLE MATTER.

Water-Soluble Matter.

53. Weigh 2.5 g. of the pigment into a graduated 250-cc. flask, add 100 cc. of water, and boil for five minutes. Dilute with water, let stand until at room temperature, make up to the mark, mix, and let settle. Filter through dry paper and discard the first 25 cc. Transfer 100 cc. of the clear filtrate to a weighed dish, evaporate to dryness on a steam-bath, dry in an oven at 105° C. for 30 minutes, cool and weigh.

TINTING STRENGTH AND ADDED COLORING MATTER

Coloring Matter.

54. Make the test for added coloring matter and tinting strength in accordance with Sections 2 and 3. Also test the pigment with dilute acid for the presence of ultramarine blue (evolution of hydrogen sulfide).

STANDARD METHODS OF ROUTINE ANALYSIS OF TITANIUM PIGMENTS[1]

Serial Designation: D 186 - 27

These methods are issued under the fixed designation D 186; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1924; ADOPTED, 1925; REVISED, 1927.

GENERAL METHODS

SPECIFIC GRAVITY

Specific Gravity.

1. True specific gravity shall be determined in accordance with the Standard Methods of Test for Specific Gravity of Pigments (Serial Designation: D 153) of the American Society for Testing Materials.[2]

COLOR

Color.

2. To 5 g. of the sample add 1.5 cc. of linseed oil, rub up on a stone slab or glass plate with a flat-bottomed glass or stone pestle or muller to a uniform smooth paste. Treat in a similar manner 5 g. of the standard titanium pigment. Spread the two pastes side by side on a clear, colorless glass plate and compare the colors. If the sample is as white as, or whiter than the "standard," it passes this test.

COLOR STRENGTH

Color Strength.

3. Weigh accurately 0.01 g. of lampblack, place on a large glass plate or stone slab, add 0.2 cc. of linseed oil and rub up with a flat-bottomed glass pestle or muller, then add exactly 10 g. of the sample and 2.5 cc. of linseed oil, and grind with a circular motion of the pestle or muller 50 times; gather up with a sharp-edged spatula and grind out twice more in a like manner, giving the pestle or muller a uniform pressure. Treat another 0.01 g. of lampblack in the same manner except that 10 g. of standard pigment is used instead of the 10 g. of the sample. Spread the two pastes side by side on a glass microscope slide and compare the colors. If the sample is as light or lighter in color than the "standard" it passes this test.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

[2] See p. 277.

COARSE PARTICLES

Coarse Particles.

4. Determine coarse particles in accordance with the Standard Method of Test for Coarse Particles in Paint Pigments (Serial Designation: D 185) of the American Society for Testing Materials.[1]

METHODS OF ANALYSIS

QUALITATIVE ANALYSIS

Qualitative Analysis.

5. Place about 0.5 g. of the sample in a 250-cc. Pyrex glass beaker; add 20 cc. of concentrated H_2SO_4 and 7 to 8 g. of $(NH_4)_2SO_4$. Mix well and boil for a few minutes. The sample should go completely into solution; a residue denotes the presence of silica or siliceous matter. Cool the solution, dilute with 100 cc. of water, heat to boiling, let settle, filter, wash with hot 5-per-cent H_2SO_4 until free from titanium, and test the residue for lead, etc. Test the filtrate for calcium, zinc, iron, chromium, etc., by the regular methods of qualitative analysis. For the iron determination add to a portion of the filtrate 5 g. of tartaric acid, render slightly ammoniacal, pass in H_2S in excess, and digest at side of steam bath for a while. No precipitate indicates the absence of iron, nickel, cobalt, lead, copper, etc. A black precipitate readily soluble in dilute HCl denotes iron. For titanium, test a small portion of the original filtrate with hydrogen peroxide (a clear yellow-orange color should result) and another portion with metallic tin or zinc (a pale blue to violet coloration should result). Negative tests should be shown for sulfide sulfur, carbonates, and appreciable water-soluble matter.

MOISTURE

Moisture.

6. Place 1 g. of the sample in a wide-mouthed short weighing tube provided with a glass stopper. Heat with stopper removed for two hours at a temperature between 105 and 110° C. Insert stopper, cool, and weigh. The loss in weight is reported as moisture.

MATTER SOLUBLE IN WATER

Matter Soluble in Water.

7. Transfer 2.5 g. of the pigment to a graduated 250-cc. flask, add 100 cc. of water, boil for 5 minutes, cool, fill to mark with water, mix, and allow to settle. Pour the supernatant liquid through a dry filter paper and discard the first 20 cc. Then evaporate 100 cc. of the clear filtrate to dryness in a weighed dish, heat for one hour at 105 to 110° C., cool and weigh.

[1] See p. 286.

TITANIUM OXIDE

Titanium Oxide.

8. Transfer 0.5 g. of the dried sample to a 250-cc. Pyrex beaker, add 20 cc. of concentrated H_2SO_4 and 7 to 8 g. of ammonium sulfate. Mix well and heat on hot plate until fumes of sulfuric acid are evolved, and then continue the heating over a strong flame until solution is complete (usually requires not over five minutes of boiling) or it is apparent that the residue is composed of silica or siliceous matter. Caution should be observed in visually examining this hot solution. Cool the solution, dilute with 100 cc. of water, stir, heat carefully to boiling while stirring, let settle, filter through paper and transfer the precipitate completely to the paper. Wash the insoluble residue with cold 5-per-cent (by volume) H_2SO_4 until titanium is removed.

Dilute the filtrate to 200 cc. and add about 10 cc. of NH_4OH (sp. gr. 0.90) to lower the acidity to approximately 5-per-cent H_2SO_4 (by volume).

Wash out a Jones reductor[1] with dilute 5 per cent by volume H_2SO_4 and water, leaving sufficient water in the reductor to fill to the upper level of the zinc. (These washings should require not more than one or two drops of 0.1 *N* $KMnO_4$ solution to obtain a pink color.) Empty the receiver, and put in it 25 cc. (measured in a graduate) of ferric sulfate solution (see Reagents). Reduce the prepared titanium solution as follows:[2]

(1) Run 50 cc. of the 5-per-cent H_2SO_4 solution through the reductor at a speed of about 100 cc. per minute.

(2) Folllow this with the titanium solution.

(3) Wash out with 100 cc. of 5 per cent H_2SO_4.

(4) Finally run through about 100 cc. of water. Care should be taken that the reductor is always filled with solution or water to the upper level of the zinc. Gradually release the suction, wash thoroughly the glass tube that was immersed in the ferric sulfate solution, remove the receiver, and titrate immediately with 0.1 *N* $KMnO_4$ solution. One cc. of 0.1 *N* $KMnO_4$ equals 0.0048 g. Ti or 0.008 g. TiO_2. Run a blank determination, using the same reagents, washing the reductor as in the above determination. Subtract this permanganate reading from the original reading and calculate the final reading to titanium dioxide (TiO_2). This will include iron, chromium, arsenic, and any other substance which is reduced by zinc and acid. See Calculations below for reporting TiO_2.

[1] Directions for preparing a Jones reductor may be found in Blair, "The Chemical Analysis of Iron," Eighth Edition, pp. 88–89, or Treadwell-Hall, "Analytical Chemistry," Vol. 2, Fifth Edition.

[2] Lundell and Knowles, "The Determination of Titanium by Reduction with Zinc and Titration with Permanganate," *Journal*, Am. Chemical Soc., Vol. 45, p. 2620 (1923).

BARIUM SULFATE

Barium Sulfate.

9. Ignite, cool, and weigh the precipitate of $BaSO_4$ obtained in separating the titanium in Section 8.

NOTE.—If sample is impure it may be necessary to purify this precipitate, using appropriate methods.

IRON OXIDE

Iron Oxide.

10. Prepare a standard ferric solution containing 0.00001 g. of Fe per cubic centimeter (see Reagents). Weigh a 1-g. portion of the sample and treat as in Section 8. Transfer without filtering to a graduated 200-cc. flask, cool, fill to the mark with water, mix, let settle, and determine iron colorimetrically as follows: Filter through a dry filter paper, discarding the first 20 cc.; transfer 50 cc. of the clear filtrate to a clean 100-cc. Nessler tube or other comparator. Add a drop or two of 0.1 *N* $KMnO_4$ solution to oxidize any ferrous iron. The faint pink color should persist for at least 5 minutes. Add 10 cc. of KCNS or $(NH_4)CNS$ solution (see Reagents), dilute with water to 100 cc., and mix thoroughly. Compare the color immediately with a series of standards, prepared side by side with the sample, in similar tubes. Prepare the standards from the standard ferric solution so as to have a range of from 0.000005 g. Fe to 0.00004 g. Fe (0.5 to 4.0 cc.). Transfer the desired volumes of the standard ferric solution to 100-cc. Nessler tubes containing 50 cc. each of an acid solution (made up by dissolving 8 g. of $(NH_4)_2SO_4$ in water, adding 20 cc. of concentrated H_2SO_4, cooling, diluting with water to 200 cc., and mixing), add a drop of 0.1 *N* $KMnO_4$ solution (or sufficient to yield a pink color that will persist for 5 minutes), and then 10 cc. of the thiocyanate solution. Finally dilute all standards with water to 100 cc. and mix each thoroughly.

NOTE.—For a single sample it is more convenient to run the standard Fe solution from a burette into a Nessler tube, containing 50 cc. of acid solution (made by dissolving 8 g. of $(NH_4)_2SO_4$ in water, adding 20 cc. of concentrated H_2SO_4, cooling and diluting with water to 200 cc., and mixing), a drop of 0.1 *N* $KMnO_4$ solution, 10 cc. of the thiocyanate solution, and then dilute with distilled water until the depth of the color produced after diluting 100 cc. and mixing, exactly matches that of the sample. From the burette reading calculate the amount of Fe. When using standards, the color comparisons must be made immediately.

CALCULATIONS

Calculations.

11. Calculate the total iron found to Fe_2O_3 and report as such. Calculate the TiO_2 equivalent by multiplying the Fe_2O_3 result by the factor 1.003 and subtract this figure from the total TiO_2 as determined in Section 8 and report the remainder as TiO_2.

Report all results on the dry or moisture-free basis.

SOLUTION REQUIRED

Solutions Required.

12. *Decinormal Potassium Permanganate Solution.*—Dissolve 3.2 g. of pure $KMnO_4$ in 1 liter of distilled water, let stand 8 to 14 days, siphon off the clear solution (or filter through asbestos), and standardize as follows: In a 400-cc. beaker dissolve 0.25 to 0.30 g. (accurately weighed) of U. S. Bureau of Standards' sodium oxalate in 250 cc. of hot water (80 to 90° C.) and add 15 cc. of dilute H_2SO_4 (1:1). Titrate at once with the $KMnO_4$ solution, *stirring the liquid vigorously and continuously.* The $KMnO_4$ must not be added more rapidly than 10 to 15 cc. per minute, and the last 0.5 to 1 cc. must be added dropwise with particular care to allow each drop to be fully decolorized before the next is introduced. The solution should not be below 60° C. by the time the end point is reached. (More rapid cooling may be prevented by allowing the beaker to stand on a small asbestos-covered hot plate during the titration. The use of a small thermometer as a stirring rod is most convenient.) The weight of sodium oxalate used multiplied by 0.833 gives its iron equivalent, or multiplied by 1.195 gives its titanium dioxide equivalent. The $KMnO_4$ solution should be kept in a glass-stoppered bottle painted black to keep out light.

Ferric Sulfate Solution for Titanium.—A solution containing 2 per cent of iron as ferric sulfate is desired and may be prepared as follows: Dissolve 20 g. of pure iron or plain carbon steel in a slight excess of HCl, oxidize with HNO_3, add about 80 cc. of H_2SO_4 and heat until fumes of the latter are evolved. Cool, dilute with water to 1000 cc., digest on a steam bath until sulfates are dissolved, and filter if necessary. Add 0.1 *N* $KMnO_4$ solution until a faint pink color persists for 5 minutes (to oxidize any ferrous iron that may be present).

Ferric ammonium sulfate may be used also.[1]

Standard Ferric Sulfate Solution for Colorimetric Determination of Iron.—Determine the strength of the ferric solution for the TiO_2 determination in terms of Fe and dilute a portion of this solution until one is obtained of the strength 1 cc. equals 0.00001 g. Fe.

Potassium Thiocyanate Indicator.—Prepare a 2-per-cent solution of the pure salt in distilled water.

[1] Gooch, "Methods in Chemical Analysis," First Edition, p. 436.

STANDARD DEFINITIONS
OF
TERMS RELATING TO PAINT SPECIFICATIONS[1]

Serial Designation: D 16 - 24

These definitions are issued under the fixed designation D 16; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1915; REVISED, 1922, 1924.

Standard.—Materials, methods, qualities, properties, etc., set forth by specification as a basis for the measurement of requirements.

Equal To.—The use of this term should be avoided if possible.

The avoidance of this term is recommended wherever possible because the specifications themselves should state the qualities, etc., desired. In specifications having the sanction of the American Society for Testing Materials, it is to be assumed that this feature will be developed to its fullest extent.

Pure.—Free from admixture of any foreign substance.

Commercially Pure.—The use of this term should be avoided if possible.

The avoidance of this term is recommended wherever possible because it involves the acceptance of standards likely to cause dispute, whereas specifications having the sanction of the American Society for Testing Materials should involve the establishment of their own standards.

Adulteration.—The partial substitution of one substance for another without acknowledgment.

The addition of the words "without acknowledgment" makes this definition clear. Substitution with acknowledgment involves no improper motive. If it is done without acknowledgment an improper motive may be assumed.

Adulterant.—A substance substituted partially for another without acknowledgment.

Opacity.—The degree of obstruction to the transmission of visible light.

In this sense "opacity" is a relative term, it being considered that given a film sufficiently thin, in paint technology at least, there is no absolutely opaque substance.

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee D-1 on Preservative Coatings for Structural Materials.

Covering Power.—The use of this term should be avoided if possible.

This term has been used so loosely that it might mean hiding power, spreading power, or the simple property of producing a coat.

Hiding Power.—The power of a paint or paint material as used to obscure a surface painted with it.

In this definition the word "obscure" means to render invisible or to cover up a surface so that it cannot be seen.

Spreading Rate.—The rate at which a paint or paint material, as used, is brushed out to a continuous uniform film expressed in terms of the area to which a unit volume, as used, is applied.

This term must not be confused with the much-abused term "spreading power." The use of the term "spreading rate" is illustrated in the following sentence: "The paint when spread on a planished iron surface at the rate of 600 sq. ft. to the gallon will not sag or run when placed in a vertical position at 70° F."

Fineness.—The extent of sub-division of a substance as indicated under definite prescribed conditions.

Crystallin.—Having a definite structure referable to one of the crystallographic systems.

According to definition a material is not crystallin if it has not a crystallin form irrespective of the optical and other properties it may possess.

Amorphous.—Without regular or definite form.

This definition as given here has a broader meaning than it possesses when it is used in mineralogical writings. Protozoa, if of definite form, are not amorphous and may not be crystallin.

Paint.—A mixture of pigment with vehicle, intended to be spread in thin coats for decoration or protection, or both.

According to this definition a mixture of pigment and varnish is a paint, and on the other hand a solution of stains in oil or varnish, no pigment being present, is not a paint.

Size.—In the painting art, a liquid coating material, intended to close the pores, used to prepare a surface for further treatment.

It is not regarded as a finishing material.

Varnish.—A liquid coating material, containing no pigment, which flows out to a smooth coat when applied and dries to a smooth, glossy, relatively hard, permanent solid when exposed in a thin film to the air.

Some materials possessing the other characteristics, dry without the usual gloss and are termed "flat varnish."

Enamel.—A special kind of paint which flows out to a smooth coat when applied and dries to a smooth, glossy, relatively hard, permanent solid when exposed in a thin film to the air. An enamel always contains pigment and has considerable hiding power and color. Some enamels dry to a flat or eggshell finish instead of a gloss finish.

Filler.—A special kind of paint used for filling pores or other small breaks in the continuity of a surface to render it smooth preparatory to further treatment. When applied and exposed to the air, a filler should dry to a relatively hard, permanent solid capable of properly supporting subsequent coats.

Pigment.—The fine solid particles used in the preparation of paint, and substantially insoluble in the vehicle.

Asphaltic materials are not pigments except when they contain substances substantially insoluble in the vehicle in which they are used.

Toner.—An organic pigment which does not contain inorganic pigment or inorganic carrying base.

Lake.—A special type of pigment consisting essentially of an organic soluble coloring matter combined more or less definitely with an inorganic base or carrier. It is characterized generally by a bright color and a more or less pronounced translucency when made into an oil paint.

Under this term are included two (and perhaps three) types of pigment: (*a*) the older original type composed of hydrate of alumina dyed with a solution of the natural organic color, (*b*) the more modern and far more extensive type made by precipitating from solution various coal-tar colors by means of a metallic salt, tannin, or other suitable reagent, upon a base or carrier either previously prepared or coincidently formed, and (*c*) a number combining both types in varying degree, might be regarded as a third class.

Vehicle.—The liquid portion of a paint.

Here anything that is dissolved in the liquid portion of a paint is a part of the vehicle.

Volatile Thinner.—All that liquid portion of a paint, water excepted, which is volatile in a current of steam at atmospheric pressure.

Non-Volatile Vehicle.—The liquid portion of a paint excepting its volatile thinner and water.

Drying Oil.—An oil which possesses to a marked degree the property of readily taking up oxygen from the air and changing to a relatively hard, tough, elastic substance when exposed in a thin film to the air.

Semi-Drying Oil.—An oil which possesses the characteristics of a drying oil but to a less degree.

There is no definite line of demarcation between drying and semi-drying oils.

Non-Drying Oil.—An oil which does not of itself possess to a perceptible degree the power to take up oxygen from the air and lose its liquid characteristics.

Tinting Strength.—The power of coloring a given quantity of paint or pigment selected as a medium standard for estimating such power.

Color.—A generic term referring inclusively to all of the colors of the spectrum, white and black, and all tints, shades and hues which may be produced by their admixture.

Color involves a definite effect produced by the action of light upon the retina of the eye dependent upon the optical composition of the light. This term is also used in reference to material substances such as pigments, stains, dyes, etc., but in specifications it should be recognized that color is primarily a physiological sensation.

Tint.—A color produced by the admixture of a coloring material, not white, with a white pigment or paint, the white predominating.

Shade.—A term descriptive of that difference between colors which results from a difference in luminosity only, the other color constants being essentially equal. A darker shade of a color is one that has a lower luminosity.

Primarily the term "shade" is akin to shadow designating darkness or reduced illumination, and therefore when strictly used should express only such change as depends on *reduced* luminosity; it has been defined by several authorities as the mixture of black with a color, thus establishing its opposite character to "tint," but by extension of its relative sense it has been frequently and widely used to include lighter shades by use of the adjective "lighter" or "paler." Although such expressions apparently involve a contradiction, it is clear that while we may have a shade of color or darker color of the same sort, it is easy to conceive of another shade not quite so dark and therefore lighter.

Hue.—The predominating spectral color in a color mixture.

Tone.—The color which principally modifies a hue or a white or a black.

Drying.—The solidification of a film.

Drier.—A material containing metallic compounds added to paints and painting materials for the purpose of accelerating drying.

Specific Gravity.—The ratio of the weight of a unit volume of a substance to the weight of an equal volume of water at defined temperatures.

Density.—The use of this term should be avoided if possible.

Density is a scientific term meaning the mass of a unit volume. Its numerical expression will vary with the units selected, and there is no occasion for using it when the term "specific gravity" is defined. Confusion may be avoided by not using the word "density" in specifications.

Gallon.—The measured gallon is 231 cu. in. Where a measured gallon is called for, the temperature at which it is to be measured should be specified. Where a gallon of definite weight is called for, the weight should be specified or obtained from the specific gravity of the material at a definite temperature.

This is the standard United States gallon.

Water.—Dissolved water or water not definitely or chemically combined.

Dry.—In paint materials: containing no uncombined water. In paint films: completely solidified.

STANDARD ABRIDGED VOLUME CORRECTION TABLE FOR PETROLEUM OILS[1]

Serial Designation: D 206 – 25

This table is issued under the fixed designation D 206; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1925.

This abridged table has been prepared by the U. S. Bureau of Standards to meet a demand from the oil industry for a short and convenient table for reducing oil volumes to the basis of 60° F. (15.5° C.). It is based on the same data as Table 2, Bureau of Standards *Circular No. 154*. The Groups, Coefficients of Expansion, Degrees A.P.I. and Gravity Ranges of the abridged table follow:[2]

Group Number	Coefficient of Expansion at 60° F.	Corresponding Degrees A. P. I.	Range of Group (degrees A. P. I. at 60° F.)
I	0.0004	22	Up to 34.9
II	0.0005	44	35 to 50.9
III	0.0006	58	51 to 63.9
IV	0.0007	72	64 to 78.9
V	0.0008	86	79 to 88.9
VI	0.00085	91	89 and higher

The table shows the volume occupied by 60° F. by a quantity of oil occupying a unit volume at the indicated temperatures. For example, if at 60° F. the A. P. I. gravity of the oil is 28 (Group I), one gallon of this oil measured at 120° F. will have a volume of 0.9763 gal. at 60° F. The values given in the table are in the form of "multipliers"; that is, the volume of oil at the indicated temperature and degrees A. P. I. for each group, multiplied by the corresponding factor in the table, equals the volume at 60° F. For example, if the A. P. I. gravity of an oil at 60° F. equals 28 (Group I) and the volume at 120° F. equals 6000 gal., then the volume at 60° F. equals 6000 × 0.9763, or 5857.8 gal.

[1] Under the standardization procedure of the Society, this table is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

The table has been approved by the Bureau of Standards, the Bureau of Mines and the American Petroleum Institute and is now published as a standard of the Society.

[2] This abridged table is not intended to replace the more complete and accurate volume correction table contained in Bureau of Standards *Circular No. 154*, but rather to supplement it and especially to replace the various abridged tables and approximate correction factors heretofore employed in the oil industry. In case high accuracy is essential the complete table contained in *Circular No. 154* should be used, especially if large volumes or wide temperature ranges are involved The A.S.T.M. Standard (Serial Designation: D 206 – 25) applies only to the Abridged Table and should be so understood when written into contracts.

STANDARD ABRIDGED VOLUME CORRECTION TABLE FOR PETROLEUM OILS.

Observed Temperature, deg. Fahr.	Volume at 60° F. Occupied by Unit Volume at Indicated Temperature					
	Group I, Up to 34.9° A.P.I. at 60° F.	Group II, 35.0 to 50.9° A.P.I. at 60° F.	Group III, 51.0 to 63.9° A.P.I. at 60° F.	Group IV, 64.0 to 78.9° A.P.I. at 60° F.	Group V, 79.0 to 88.9° A.P.I at 60° F.	Group VI, 89.0° and higher, A.P.I. at 60° F.
0	1.0242	1.0297	1.0361	1.0419	1.0476	1.0501
1	1.0238	1.0292	1.0355	1.0412	1.0468	1.0493
2	1.0234	1.0287	1.0349	1.0405	1.0460	1.0484
3	1.0230	1.0283	1.0343	1.0398	1.0453	1.0476
4	1.0226	1.0278	1.0337	1.0391	1.0445	1.0467
5	1.0222	1.0273	1.0331	1.0384	1.0437	1.0459
6	1.0218	1.0268	1.0325	1.0377	1.0429	1.0451
7	1.0214	1.0263	1.0319	1.0370	1.0421	1.0443
8	1.0210	1.0258	1.0313	1.0364	1.0414	1.0434
9	1.0206	1.0253	1.0307	1.0357	1.0406	1.0426
10	1.0202	1.0248	1.0301	1.0350	1.0398	1.0418
11	1.0198	1.0243	1.0295	1.0343	1.0390	1.0410
12	1.0194	1.0238	1.0289	1.0336	1.0382	1.0402
13	1.0189	1.0233	1.0283	1.0329	1.0375	1.0393
14	1.0185	1.0228	1.0277	1.0322	1.0367	1.0385
15	1.0181	1.0223	1.0271	1.0315	1.0359	1.0377
16	1.0177	1.0218	1.0265	1.0308	1.0351	1.0369
17	1.0173	1.0213	1.0259	1.0301	1.0343	1.0360
18	1.0169	1.0208	1.0253	1.0294	1.0335	1.0352
19	1.0165	1.0203	1.0247	1.0287	1.0327	1.0343
20	1.0161	1.0198	1.0241	1.0280	1.0319	1.0335
21	1.0157	1.0193	1.0235	1.0273	1.0311	1.0327
22	1.0153	1.0188	1.0229	1.0266	1.0303	1.0319
23	1.0148	1.0184	1.0223	1.0260	1.0296	1.0310
24	1.0144	1.0179	1.0217	1.0253	1.0288	1.0302
25	1.0140	1.0174	1.0211	1.0246	1.0280	1.0294
26	1.0136	1.0169	1.0205	1.0239	1.0272	1.0286
27	1.0132	1.0164	1.0199	1.0232	1.0264	1.0277
28	1.0128	1.0159	1.0193	1.0225	1.0256	1.0269
29	1.0124	1.0154	1.0187	1.0218	1.0248	1.0260
30	1.0120	1.0149	1.0181	1.0211	1.0240	1.0252
31	1.0116	1.0144	1.0175	1.0204	1.0232	1.0244
32	1.0112	1.0139	1.0169	1.0197	1.0224	1.0236
33	1.0108	1.0134	1.0163	1.0190	1.0216	1.0227
34	1.0104	1.0129	1.0157	1.0183	1.0208	1.0219
35	1.0100	1.0124	1.0151	1.0176	1.0200	1.0211
36	1.0096	1.0119	1.0145	1.0169	1.0192	1.0203
37	1.0092	1.0114	1.0139	1.0162	1.0184	1.0194
38	1.0088	1.0109	1.0133	1.0155	1.0176	1.0186
39	1.0084	1.0104	1.0127	1.0148	1.0168	1.0177
40	1.0080	1.0099	1.0121	1.0141	1.0160	1.0169
41	1.0076	1.0094	1.0115	1.0134	1.0152	1.0160
42	1.0072	1.0089	1.0109	1.0127	1.0144	1.0152
43	1.0068	1.0084	1.0103	1.0120	1.0136	1.0143
44	1.0064	1.0079	1.0097	1.0113	1.0128	1.0135
45	1.0060	1.0074	1.0091	1.0106	1.0120	1.0126
46	1.0056	1.0069	1.0085	1.0099	1.0112	1.0118
47	1.0052	1.0064	1.0079	1.0092	1.0104	1.0110
48	1.0048	1.0059	1.0072	1.0084	1.0096	1.0101
49	1.0044	1.0054	1.0066	1.0077	1.0088	1.0093
50	1.0040	1.0049	1.0060	1.0070	1.0080	1.0084
51	1.0036	1.0044	1.0054	1.0063	1.0072	1.0076
52	1.0032	1.0039	1.0048	1.0056	1.0064	1.0067
53	1.0028	1.0035	1.0042	1.0049	1.0056	1.0059
54	1.0024	1.0030	1.0036	1.0042	1.0048	1.0050
55	1.0020	1.0025	1.0030	1.0035	1.0040	1.0042
56	1.0016	1.0020	1.0024	1.0028	1.0032	1.0034
57	1.0012	1.0015	1.0018	1.0021	1.0024	1.0025
58	1.0008	1.0010	1.0012	1.0014	1.0016	1.0017
59	1.0004	1.0005	1.0006	1.0007	1.0008	1.0008

STANDARD ABRIDGED VOLUME CORRECTION TABLE FOR PETROLEUM OILS (*Continued*).

Observed Temperature, deg. Fahr.	Volume at 60° F. Occupied by Unit Value at Indicated Temperature					
	Group I, Up to 34.9° A.P.I. at 60° F.	Group II, 35.0 to 50.9° A.P.I. at 60° F.	Group III, 51.0 to 63.9° A.P.I. at 60° F.	Group IV, 64.0 to 78.9° A.P.I. at 60° F.	Group V, 79.0 to 88.9° A.P.I. at 60° F.	Group VI, 89.0° and higher, A.P.I. at 60° F.
60	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
61	0.9996	0.9995	0.9994	0.9993	0.9992	0.9992
62	0.9992	0.9990	0.9988	0.9986	0.9984	0.9983
63	0.9988	0.9985	0.9982	0.9979	0.9976	0.9975
64	0.9984	0.9980	0.9976	0.9972	0.9968	0.9966
65	0.9980	0.9975	0.9970	0.9965	0.9960	0.9958
66	0.9976	0.9970	0.9964	0.9958	0.9952	0.9949
67	0.9972	0.9965	0.9957	0.9951	0.9944	0.9941
68	0.9968	0.9961	0.9951	0.9943	0.9935	0.9932
69	0.9964	0.9956	0.9945	0.9936	0.9927	0.9924
70	0.9960	0.9951	0.9939	0.9929	0.9919	0.9915
71	0.9956	0.9946	0.9933	0.9922	0.9911	0.9907
72	0.9952	0.9941	0.9927	0.9915	0.9903	0.9898
73	0.9948	0.9935	0.9921	0.9908	0.9895	0.9890
74	0.9944	0.9930	0.9915	0.9901	0.9887	0.9881
75	0.9940	0.9925	0.9909	0.9894	0.9879	0.9873
76	0.9936	0.9920	0.9903	0.9887	0.9871	0.9864
77	0.9932	0.9915	0.9897	0.9880	0.9863	0.9856
78	0.9929	0.9911	0.9891	0.9872	0.9854	0.9847
79	0.9925	0.9906	0.9885	0.9865	0.9846	0.9839
80	0.9921	0.9901	0.9879	0.9858	0.9838	0.9830
81	0.9917	0.9896	0.9873	0.9851	0.9830	0.9821
82	0.9913	0.9891	0.9867	0.9844	0.9822	0.9813
83	0.9909	0.9886	0.9860	0.9837	0.9814	0.9804
84	0.9905	0.9881	0.9854	0.9830	0.9806	0.9796
85	0.9901	0.9876	0.9848	0.9823	0.9798	0.9787
86	0.9897	0.9871	0.9842	0.9816	0.9790	0.9779
87	0.9893	0.9866	0.9836	0.9809	0.9782	0.9770
88	0.9889	0.9861	0.9830	0.9802	0.9773	0.9762
89	0.9885	0.9856	0.9824	0.9795	0.9765	0.9753
90	0.9881	0.9851	0.9818	0.9788	0.9757	0.9745
91	0.9877	0.9846	0.9812	.....	.....	.....
92	0.9873	0.9841	0.9806	.....	.....	.....
93	0.9869	0.9836	0.9800	.....	.....	.....
94	0.9865	0.9831	0.9794	.....	.....	.....
95	0.9861	0.9826	0.9788	.....	.....	.....
96	0.9857	0.9821	0.9782	.....	.....	.....
97	0.9853	0.9816	0.9776	.....	.....	.....
98	0.9849	0.9812	0.9769	.....	.....	.....
99	0.9845	0.9807	0.9763	.....	.....	.....
100	0.9841	0.9802	0.9757	.....	.....	.....
101	0.9837	0.9797	0.9751	.....	.....	.....
102	0.9833	0.9792	0.9745	.....	.....	.....
103	0.9830	0.9786	0.9738	.....	.....	.....
104	0.9826	0.9781	0.9732	.....	.....	.....
105	0.9822	0.9776	0.9726	.....	.....	.....
106	0.9818	0.9771	0.9720	.....	.....	.....
107	0.9814	0.9766	0.9714	.....	.....	.....
108	0.9811	0.9762	0.9708	.....	.....	.....
109	0.9807	0.9757	0.9702	.....	.....	.....
110	0.9803	0.9752	0.9696	.....	.....	.....
111	0.9799	0.9747	0.9690	.....	.....	.....
112	0.9795	0.9742	0.9684	.....	.....	.....
113	0.9791	0.9737	0.9678	.....	.....	.....
114	0.9787	0.9732	0.9672	.....	.....	.....
115	0.9783	0.9727	0.9666	.....	.....	.....
116	0.9778	0.9722	0.9660	.....	.....	.....
117	0.9775	0.9717	0.9654	.....	.....	.....
118	0.9771	0.9712	0.9647	.....	.....	.....
119	0.9767	0.9707	0.9641	.....	.....	.....

STANDARD ABRIDGED VOLUME CORRECTION TABLE FOR PETROLEUM OILS (*Continued*).

Observed Temperature, deg. Fahr.	Volume at 60° F. Occupied by Unit Volume at Indicated Temperature			Observed Temperature, deg. Fahr.	Volume at 60° F. Occupied by Unit Volume at Indicated Temperature		Observed Temperature, deg. Fahr.	Volume at 60° F. Occupied by Unit Volume at Indicated Temperature
	Group I, Up to 34.9° A.P.I. at 60° F.	Group II, 35.0 to 50.9° A.P.I. at 60° F.	Group III, 51.0 to 63.9° A.P.I. at 60° F.		Group I, Up to 34.9° A.P.I. at 60° F.	Group II, 35.0 to 50.9° A.P.I. at 60° F.		Group I, Up to 34.9° A.P.I. at 60°F.
120	0.9763	0.9702	0.9635	165	0.9590	.....	210	0.9419
121	0.9759	0.9697	0.9629	166	0.9586	.....	211	0.9415
122	0.9755	0.9692	0.9623	167	0.9582	.....	212	0.9412
123	0.9752	0.9687	0.9617	168	0.9578	.....	213	0.9408
124	0.9748	0.9682	0.9611	169	0.9574	.....	214	0.9405
125	0.9744	0.9677	0.9605	170	0.9570	.....	215	0.9401
126	0.9740	0.9672	.....	171	0.9566	.....	216	0.9397
127	0.9736	0.9667	.....	172	0.9562	.....	217	0.9393
128	0.9732	0.9662	.....	173	0.9559	.....	218	0.9390
129	0.9728	0.9657	.....	174	0.9555	.....	219	0.9386
130	0.9724	0.9652	.....	175	0.9551	.....	220	0.9382
131	0.9720	0.9647	.....	176	0.9547	.....	221	0.9378
132	0.9716	0.9642	.....	177	0.9543	.....	222	0.9374
133	0.9713	0.9637	.....	178	0.9540	.....	223	0.9371
134	0.9709	0.9632	.....	179	0.9536	.....	224	0.9367
135	0.9705	0.9627	.....	180	0.9532	.....	225	0.9363
136	0.9701	0.9622	.....	181	0.9528	.....	226	0.9359
137	0.9697	0.9617	.....	182	0.9524	.....	227	0.9356
138	0.9694	0.9613	.....	183	0.9521	.....	228	0.9352
139	0.9690	0.9608	.....	184	0.9517	.....	229	0.9349
140	0.9686	0.9603	.....	185	0.9513	.....	230	0.9345
141	0.9682	0.9598	.....	186	0.9509	.....	231	0.9341
142	0.9678	0.9593	.....	187	0.9505	.....	232	0.9337
143	0.9675	0.9588	.....	188	0.9502	.....	233	0.9334
144	0.9671	0.9583	.....	189	0.9498	.....	234	0.9330
145	0.9667	0.9578	.....	190	0.9494	.....	235	0.9326
146	0.9663	0.9573	.....	191	0.9490	.....	236	0.9322
147	0.9659	0.9568	.....	192	0.9487	.....	237	0.9318
148	0.9655	0.9563	.....	193	0.9483	.....	238	0.9315
149	0.9651	0.9558	.....	194	0.9480	.....	239	0.9311
150	0.9647	0.9553	.....	195	0.9476	.....	240	0.9307
151	0.9643	.....	.....	196	0.9472	.....	241	0.9303
152	0.9639	.....	.....	197	0.9468	.....	242	0.9300
153	0.9636	.....	.....	198	0.9465	.....	243	0.9296
154	0.9632	.....	.....	199	0.9461	.....	244	0.9293
155	0.9628	.....	.....	200	0.9457	.....	245	0.9289
156	0.9624	.....	.....	201	0.9453	.....	246	0.9285
157	0.9620	.....	.....	202	0.9449	.....	247	0.9281
158	0.9616	.....	.....	203	0.9446	.....	248	0.9278
159	0.9612	.....	.....	204	0.9442	.....	249	0.9274
160	0.9608	.....	.....	205	0.9438	.....	250	0.9270
161	0.9604	.....	.....	206	0.9434	.....		
162	0.9601	.....	.....	207	0.9430	.....		
163	0.9597	.....	.....	208	0.9427	.....		
164	0.9594	.....	.....	209	0.9423	.....		

NOTE.—For temperatures above 203° F. the tabulated values of the volume at 60° F. are extrapolated from the experimental results obtained over the temperature range 32 to 203° F.

STANDARD METHOD OF ANALYSIS OF GREASE[1]

Serial Designation: D 128 – 27

This method is issued under the fixed designation D 128; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED IN AMENDED FORM, 1927.

Scope.

1. These methods of analysis permit determinations sufficiently accurate for referee purposes of all the constituents of greases likely to be covered by specifications. These constituents are fillers and ash, soap bases, soap, fat, water, excess alkali or acid, petroleum products and unsaponifiable matter. No quantitative determination of glycerin is described, but a procedure is outlined for its qualitative detection.

SAMPLE

Sample.

2. The size of sample for the soap determination (Methods (1) and (2) for Determination of Fillers, Soap, Fat, Petroleum Oil and Unsaponifiable Matter) shall be from about 8 to 30 g., depending on the consistency of the grease, which is chiefly determined by the percentage of soap present. Ten to twenty grams is usually a convenient amount for No. 3 Cup Grease, while thin transmission and other greases require a larger sample. The original sample should be stirred or mixed until uniform.

Accuracy of Weighing.

3. Samples need not be weighed more closely than 0.1 g.

SOLVENTS

Petroleum Ether.

4. The petroleum ether shall have an end point not higher than 200° F., and 125 cc. of it shall not consume more than 0.2 cc. of 0.5 *N* potassium hydroxide when a blank test is made as follows:

One hundred and twenty-five cubic centimeters of petroleum ether shall be boiled with 10 cc. of 0.5 *N* alcoholic potassium hydroxide and 50 cc. of neutral 50-per-cent alcohol for $1\frac{1}{2}$ hours on a hot plate, using a glass tube of about 7 mm. internal diameter and 75 cm. long as a reflux condenser. After cooling, the solutions shall be titrated with 0.5 *N* hydrochloric acid and phenolphthalein. Not

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

less than 9.8 cc. of 0.5 *N* acid shall be required for neutralization. The amount of alkali consumed in this test shall be deducted as a blank correction in the fat determination on solution (*E*).[1]

Alcohol.

5. The alcohol shall be prepared from commercial 95-per-cent grain or denatured grain alcohol by distilling from sodium hydroxide, diluting to 50 per cent (or 70 per cent) by volume with distilled water and neutralizing exactly with sodium or potassium hydroxide and phenolphthalein.

Phenolphthalein.

6. The phenolphthalein solution shall be prepared by dissolving 1.0 g. of phenolphthalein in 50 cc. of strong redistilled alcohol, adding 50 cc. of water, and neutralizing with sodium or potassium hydroxide.

Hydrochloric Acid.

7. Concentrated c.p. hydrochloric acid shall be used when "concentrated hydrochloric acid" is specified. Hydrochloric acid, 10-per-cent, shall contain 10 per cent by weight of absolute HCl, with a permissible variation of ± 0.5 per cent.

PROCEDURE

(*A*) *Determination of Ash.*

NOTE.—The determination of the total ash should not in general be regarded as of any great importance. It is, however, sometimes required. This determination is often unsatisfactory on account of interaction between sodium carbonate derived from the soap, and inorganic fillers. There is always likelihood of reaction with the porcelain crucible itself on account of the long continued heating necessary to burn out all carbon. Moreover, if much sodium or potassium carbonate is present, the ash is fusible and often encloses carbon, making complete removal of the latter very difficult. Results will always be low in the presence of easily reducible oxides of volatile metals. There is also uncertainty as to when calcium carbonate has been completely ignited to calcium oxide. Ash determinations made on the same sample in different laboratories are likely to vary widely. For these reasons, it is usually preferable to make separate determinations of the percentage of fillers and of soap, from which data a calculation is easily made giving all the information to be gained from a direct determination of total ash.

8. The percentage of ash shall not be included in the total of the analysis.

(*1*) *Rapid Routine Method of Ash Determination.*

Ash, Routine.

9. From 2 to 5 g. of grease shall be placed in a weighed porcelain crucible, and the sample weighed to the nearest 0.1 g. If lead or zinc soap is known to be absent, a platinum crucible is more convenient. The combustible matter shall be burned off slowly and the residue finally ignited until the ash is free of carbonaceous matter. The crucible and contents shall then be cooled in a desiccator and weighed, reporting the result as percentage of ash.

[1] Solutions designated by capital letters in parentheses refer to the solutions given in Fig. 1.

(2) Alternative Method of Ash Determination.

Ash, Alternative.

10. The sample shall be burned as in method (1) until the ash is nearly free from carbon. The crucible and contents shall then be cooled, the soluble portions dissolved in a little water, and a slight excess of dilute sulfuric acid added, running the acid in carefully from a pipette inserted under a small watch glass covering the crucible. The crucible and contents shall then be warmed on the steam bath until effervescence has ceased. The watch glass shall then be rinsed with water into the crucible. The solution when tested with methyl orange for free acidity shall show free acid present. The contents of the crucible shall be evaporated to dryness, and the whole ignited at a low red heat, adding a few small pieces of dry ammonium carbonate to drive off the excess of sulfur trioxide. After cooling and weighing, the result shall be reported as "percentage of ash as sulfates."

The alternative method gives more concordant results than the first method, but requires more time and manipulation.

(B) Qualitative Examination of Ash.

Detection of Bases.

11. An easily fusible ash, dissolving completely in water to give a strongly alkaline solution, indicates a grease containing sodium, potassium, or both. A white infusible ash, practically insoluble in water, but imparting to it an alkaline reaction, may indicate calcium, with or without magnesium or aluminium. Zinc is shown by the yellow color of the ash while hot, and lead may be indicated by the presence of metallic globules, or by the yellow color of the ash when cold.

12. The ash shall be dissolved in dilute nitric or hydrochloric acid, and the presence of the several bases confirmed by suitable chemical tests, any standard scheme of qualitative analysis being followed.

(C) Quantitative Examination of Ash.

Quantitative Examination.

13. For the quantitative examination of ash any standard analytical procedure shall be used, the choice of methods being based on the information gained from the qualitative tests. If only one base is present, a quantitative determination is in general unnecessary.

(D) Determination of Fillers, Soap, Fat, Petroleum Oil and Unsaponifiable Matter.

Choice of Method.

14. Cup, fiber and sponge greases, with or without graphite, and all comparatively light colored greases, including axle greases,

shall be examined by method (1). Dark greases containing residuum, asphaltic oils and asphalt, tars, etc., shall be examined by method (2), as these ingredients usually cause stubborn emulsions if a shaking-out process is applied at the start.

(1) Determination of Filler, Soap, Fat, Etc.

Determination of Filler

15. If no fillers are present, Sections 15 and 16 shall be passed over and the grease sample introduced directly from a weighing bottle into a separator, the weight of the sample being obtained by difference. (See Section 2.) The sample shall then be shaken with 75 cc. of petroleum ether and 50 cc. of 10-per-cent hydrochloric acid until the grease is completely decomposed, after which Section 17 shall be followed. If fillers are present, the sample (See Section 2) shall be weighed in a small beaker, 50 cc. of 10-per-cent hydrochloric acid added and the beaker warmed on the steam bath, stirring until all soap lumps have disappeared and the upper layer is clear. If undissolved mineral matter or other filler is present, both layers shall be filtered through a Gooch crucible provided with a suitable mat, the beaker and crucible washed with water and petroleum ether, and the crucible finally washed with strong alcohol, the alcohol washings being collected separately and discarded. The crucible and contents shall then be dried at 120° C. and weighed, and the result reported as "percentage of insoluble matter" (graphite, mica, talc, asbestos, gypsum, wood pulp, etc.)

Determination of Gypsum.

16. If gypsum is present as a filler, the results obtained in the determination described in Section 15 will be too low because of the solubility of calcium sulfate in hydrochloric acid. A sample of about 5 g. of grease shall be weighed in a small beaker, and decomposed with 50 cc. of petroleum ether and 25 cc. of concentrated hydrochloric acid, warming on the steam bath until all gypsum is dissolved. The contents of the beaker shall be cooled and poured into a separatory funnel, the beaker rinsed with water and a little petroleum ether and the solutions in the funnel allowed to separate clearly. The lower layer shall then be drawn into a beaker, and the petroleum ether in the separator washed with two portions of 20 cc. each of 10-per-cent hydrochloric acid, and the washes added to the strong acid solution in the beaker. The hydrochloric acid solution in the beaker shall then be evaporated on the steam bath almost to dryness, diluted to 150 cc. with water, heated to boiling and treated with 10 cc. of 10-per-cent barium chloride solution. The barium sulfate shall then be filtered off and ignited in the usual way, and from the weight of the barium sulfate, the percentage of calcium sulfate shall be calculated, reporting it as $CaSO_4 \cdot 2H_2O$.

NOTE.—Chalk and other forms of alkaline earth carbonates are not determined by this method, but are considered under the determination of free alkalinity, Section 24.

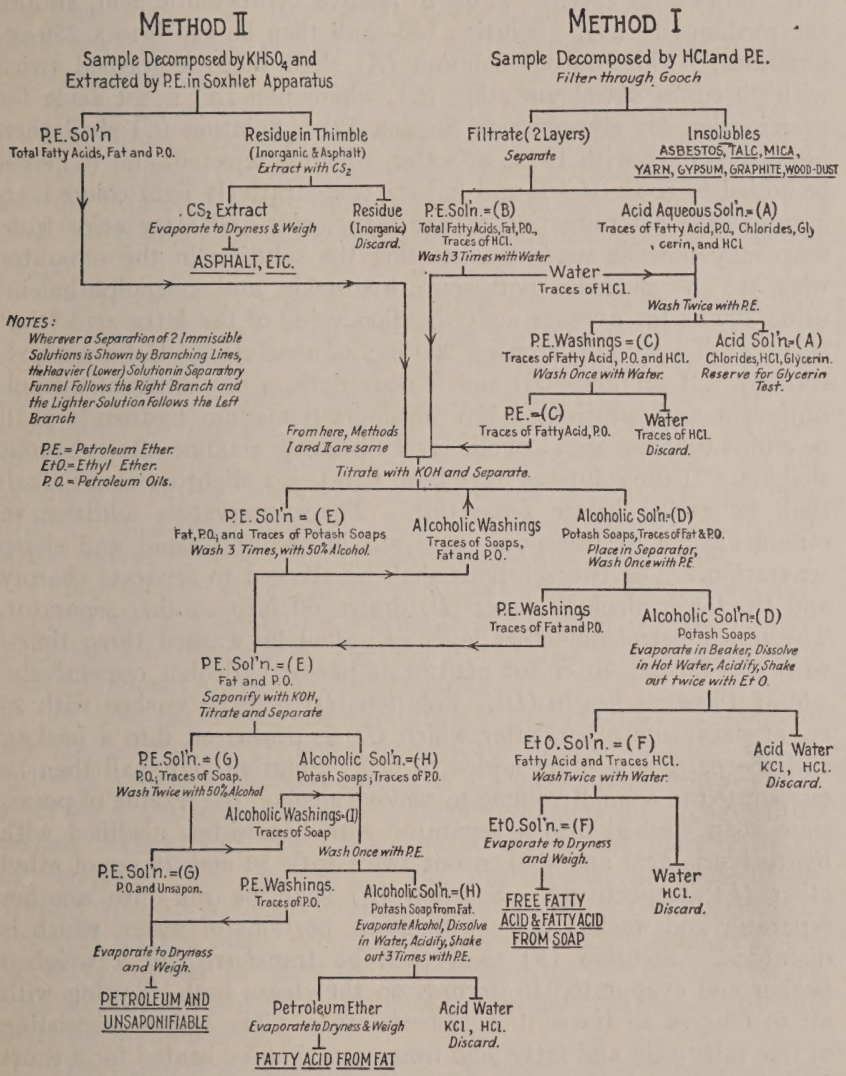

METHOD II
Sample Decomposed by $KHSO_4$ and Extracted by P.E. in Soxhlet Apparatus
P.E. Sol'n
Total Fatty Acids, Fat and P.O.
Residue in Thimble
(Inorganic & Asphalt)
Extract with CS_2
CS_2 Extract
Evaporate to Dryness & Weigh
ASPHALT, ETC.
Residue
(Inorganic)
Discard.
METHOD I
Sample Decomposed by HCl and P.E.
Filter through Gooch
Filtrate (2 Layers)
Separate
Insolubles
ASBESTOS, TALC, MICA, YARN, GYPSUM, GRAPHITE, WOOD-DUST
P.E. Soln. = (B)
Total Fatty Acids, Fat, P.O., Traces of HCl.
Wash 3 Times with Water
Acid Aqueous Sol'n = (A)
Traces of Fatty Acid, P.O., Chlorides, Glycerin, and HCl
Water
Traces of H.Cl.
Wash Twice with P.E.
NOTES:
Wherever a Separation of 2 Immiscible Solutions is Shown by Branching Lines, the Heavier (Lower) Solution in Separatory Funnel Follows the Right Branch and the Lighter Solution Follows the Left Branch
P.E. = Petroleum Ether.
Et O = Ethyl Ether.
P.O. = Petroleum Oils.
P.E. Washings = (C)
Traces of Fatty Acid, P.O. and HCl.
Wash Once with Water.
Acid Sol'n = (A)
Chlorides, HCl, Glycerin.
Reserve for Glycerin Test.
P.E. = (C)
Traces of Fatty Acid, P.O.
Water
Traces of HCl.
Discard.
From here, Methods I and II are same
Titrate with KOH and Separate.
P.E. Sol'n = (E)
Fat, P.O., and Traces of Potash Soaps
Wash 3 Times, with 50% Alcohol.
Alcoholic Washings
Traces of Soaps, Fat and P.O.
Alcoholic Sol'n = (D)
Potash Soaps, Traces of Fat & P.O.
Place in Separator, Wash Once with P.E.
P.E. Washings
Traces of Fat and P.O.
Alcoholic Sol'n = (D)
Potash Soaps
Evaporate in Beaker, Dissolve in Hot Water, Acidify, Shake out twice with Et O.
P.E. Sol'n. = (E)
Fat and P.O.
Saponify with KOH, Titrate and Separate
Et O. Sol'n. = (F)
Fatty Acid and Traces HCl.
Wash Twice with Water.
Acid Water
KCl, HCl.
Discard.
P.E. Sol'n. = (G)
P.O. and Traces of Soap.
Wash Twice with 50% Alcohol
Alcoholic Sol'n. = (H)
Potash Soaps; Traces of P.O.
Alcoholic Washings = (I)
Traces of Soap
Wash Once with P.E.
Et O. Sol'n. = (F)
Evaporate to Dryness and Weigh.
FREE FATTY ACID & FATTY ACID FROM SOAP
Water
HCl.
Discard.
P.E. Sol'n. = (G)
P.O. and Unsapon.
P.E. Washings.
Traces of P.O.
Alcoholic Sol'n. = (H)
Potash Soap from Fat.
Evaporate Alcohol, Dissolve in Water, Acidify, Shake out 3 Times with P.E.
Evaporate to Dryness and Weigh.
PETROLEUM AND UNSAPONIFIABLE
Petroleum Ether
Evaporate to Dryness & Weigh
FATTY ACID FROM FAT
Acid Water
KCl, HCl.
Discard.

FIG. 1.—Scheme of Grease Analysis.

17. The solution resulting from the decomposition of the filler-free grease or the combined filtrate and washes from the determination of fillers (Section 15), except the alcohol, which is discarded, shall be placed in a separator, allowed to clear, and the aqueous layer (*A*),[1] **Determination of Soap.**

[1] Solutions designated by capital letters in parentheses refer to the solutions given in Fig. 1.

which contains all the bases as well as glycerin, shall be drawn into another separator. The petroleum ether layer (*B*) shall be washed three times with 25cc. of water to remove hydrochloric acid, adding the washings to (*A*). Solution (*B*) shall then be run into a 250-cc. separator. The aqueous solution (*A*) shall then be washed twice with 20 cc. of petroleum ether (*C*), whereupon (*A*) is set aside for examination for glycerin (see Section 18). Solution (*C*) shall then be washed once with 15 cc. of water, which is rejected, and (*C*) shall be added to (*B*). If (*B*) and (*C*) are comparatively light colored, an approximate determination of free fatty acids and fatty acids from soap may now be made by titrating the solution in the separator with 0.5 *N* alcoholic potassium hydroxide and phenolphthalein, using 200 as the average neutralization value of the fatty acid (that is, 1.0 g. of fatty acid requires 200 mg. of absolute KOH for neutralization). If solutions (*B*) and (*C*) are dark, a few drops of phenolphthalein and sufficient 0.5 *N* alcoholic potassium hydroxide shall be added to make the alcoholic layer distinctly alkaline after vigorous shaking. If the solutions have been titrated, a slight excess of alkali shall be added before separating. The conservative addition of strong alcohol at this point may aid in securing rapid and sharp separation. The two solutions shall be allowed to separate sharply and the lower alcoholic layer (*D*) drawn off into another separator. The upper petroleum ether layer (*E*) shall be washed three times, with 30, 25 and 20 cc. of neutral 50-per-cent alcohol respectively, adding these washes to (*D*). Solution (*D*) shall be washed with 25 cc. of petroleum ether, after which (*D*) is drawn off into a beaker, and the petroleum ether added to (*E*). Solution (*D*) shall then be evaporated to a small volume to remove alcohol, the residue of potassium soap washed into a separator with hot water, acidified with hydrochloric acid and shaken out twice with 50 and 25 cc. of ethyl ether (*F*), respectively. Solution (*F*) shall be run into another separator and washed twice with 20-cc. portions of water, which is discarded. Solution (*F*) shall then be transferred to a weighed beaker and evaporated to dryness on the steam bath, blowing with air to remove all traces of petroleum ether. The residue consisting of free fatty acid and fatty acid from soap shall be heated for a short time on a steam bath, adding and evaporating 5 cc. of absolute alcohol to remove the last traces of water, and weighed. The exact neutralization value shall then be determined on as large a sample of these fatty acids as possible. From the total fatty acid found here, the free acid if any (see Sections 23 and 25) shall be deducted and the

remainder calculated to percentage of soap, the ash analysis being used as a guide in distributing the bases, if more than one are present.

The fatty acids may be identified to some extent by special tests, such as odor, crystal form, melting point; iodine number, neutralization value, color reactions, etc.

If the grease is appreciably oxidized, the fatty acids obtained by the ethyl ether extraction are likely to be dark in color and hard to identify. For further study and identification, the neutralized acids may be extracted again (qualitatively) with petroleum ether and dilute HCl, thus eliminating the small amount of more darkly colored matter.

Detection of Glycerin.

18. In case it is desired to determine whether a grease has been made from whole fats or from fatty acids, solution (*A*) shall be neutralized with dry sodium carbonate and sufficient excess added to precipitate calcium or other metals. The whole mass shall then be evaporated to dryness, the residue extracted several times with strong alcohol, the combined alcoholic extracts filtered, and the alcohol evaporated. The residue will then contain most of the glycerin, with a little sodium chloride. The presence or absence of glycerin in the residue shall be confirmed by suitable qualitative tests.

Since fats on saponification yield between 10 and 11 per cent of glycerin, and this analytical scheme does not include a quantitative determination of glycerin, the total percentages in greases made from fats should always be less than 100.[1]

Determination of Fat.

19. The petroleum ether solution (*E*) (Section 17), containing free fat, petroleum oils, and unsaponifiable matter shall be concentrated to a volume of about 125 cc. in a 300-cc. Erlenmeyer flask, 10 cc. of 0.5 *N* alcoholic potassium hydroxide and 50 cc. of strong neutral alcohol added, and the whole boiled on a hot plate with an air condenser for 1½ hours. The uncombined alkali shall be titrated with 0.5 *N* hydrochloric acid, and from the alkali consumed, corrected for the blank determination as specified in Section 4, the percentage of free fat shall be calculated, using 195 as the average saponification value (that is, 1.0 g. of fat requires 195 mg. of absolute KOH for saponification.)

The titrated solution (*G*) shall be placed in a separator, the alcoholic lower layer (*H*) drawn off into another separator and the remaining traces of soap removed as described for (*B*) and (*C*), Section 17, making only two washes with 30 and 20 cc. of 50-per-cent alcohol (*I*). Solutions (*H*) and (*I*) shall be combined, washed once

[1] A close approximation to the amount of glycerin present may be calculated and included in the analysis, by taking 11.0 per cent of the weight of the fatty acids from soaps, providing, of course, that the grease was made from neutral fats.

with a little petroleum ether, which is added to solution (*G*), and solutions (*H*) and (*I*) then evaporated to a small volume and the fatty acid isolated as described for (*D*), Section 17. The percentage of fat may be checked by weighing and titrating the free fatty acid.[1]

Determination of Petroleum Oil.

20. The petroleum ether solution (*G*), now containing all the hydrocarbon oils and unsaponifiable matter shall be evaporated to dryness in a weighed beaker as described for (*F*), Section 17, the residue weighed, and the result reported as "petroleum oils plus unsaponifiable matter."

An approximate determination of the viscosity of the petroleum oil may be made by using a 2 or 5-cc. pipette, which has been standardized against oils of known viscosities. If a complete characterization of the petroleum products is required, a new sample of from 150 to 200 g. of grease shall be decomposed as described above, except that all quantitative operations shall be omitted, as well as the isolation of the free fatty acids, and the use of standard alkali and acid is not necessary.

If the grease contains rosin oil, beeswax, de gras, spermaceti, spermatin, montan wax, candle pitch, and other materials containing a large amount of unsaponifiable substances, the petroleum oils isolated from solution (*G*) will contain the unsaponifiable matter, and the constants found will differ from those of the petroleum products used in making the grease. In most cases, no further separation is possible except in the hands of skillful and experienced operators who can devise special methods to suit the individual conditions.

(2) Determination of Filler, Soap, Fat, Etc.

Method for Dark Greases.

21. The sample shall be weighed in a 3-in. porcelain dish, 10 g. of granulated acid potassium sulfate added and 10 g. of clean dry ignited sand. The dish and contents shall be heated on the steam bath with frequent stirring until all water is driven off, two hours usually being sufficient. After cooling and breaking up lumps with a small pestle, the mixture shall be transferred quantitatively to an extraction thimble, using a little petroleum ether to wash the last traces into the thimble, which should be already placed in a Soxhlet apparatus. The thimble shall be extracted thoroughly with petroleum ether, the extract concentrated somewhat if necessary, and the free fatty acid and fatty acid from soap titrated with 0.5 *N* alcoholic potassium hydroxide as in solutions (*B*) and (*C*), in method (1), Section 17. From this point on, the procedure is identical with that given in method (1), Sections 17, 19 and 20.

[1] Multiplying the weight of fatty acid by 1.045 gives a very close approximation of the weight of fat from which it was derived. This factor varies very little with the molecular weight of the fat.

22. The thimble shall be extracted a second time with carbon disulfide, the extract evaporated to dryness, heated for one hour to 120° C. and weighed, the results being reported as asphaltic and tarry matter. The residue in the thimble shall be discarded. Determination of Asphalt and Tarry Matter.

(E) Determination of Free Alkali and Free Acid.

23. From 10 to 30 g. of grease shall be weighed in a small beaker, dissolved as completely as possible in 75 cc. of petroleum ether by stirring with a spatula, the mixture washed into a 250-cc. Erlenmeyer flask with a small amount of petroleum ether, and the beaker rinsed with 50 cc. of 50-per-cent alcohol, the alcohol being poured into the flask, after which a few drops of phenolphthalein solution shall be added and the whole shaken vigorously. If the alcoholic layer, after settling for a few seconds, is pink, 10 cc. of 0.5 *N* hydrochloric acid shall be added, the solutions boiled on a hot plate for ten minutes to expel carbon dioxide, and the excess acid titrated back with 0.5 *N* alcoholic potassium hydroxide. The free alkalinity shall be calculated in terms of hydroxide of the predominating base. Free Alkali.

24. If chalk or any other form of alkaline earth carbonate, or lead carbonate, is present as a filler, it will be detected by effervescence on adding the hydrochloric acid. As the amount of such carbonates is likely to be considerable, the volume of 0.5 *N* hydrochloric acid added shall be increased sufficiently to dissolve all carbonate and leave a slight excess of acid. The solution shall be boiled for two minutes and the excess acid titrated back with 0.5 *N* alcoholic potassium hydroxide, and from the acid consumed, its equivalent in calcium carbonate, etc., shall be calculated, disregarding any other forms of alkalinity which may have been present. Insoluble Carbonates.

25. If the original alcoholic layer is not pink, the solution shall be titrated carefully in the cold with 0.5 *N* alcoholic potassium hydroxide, shaking well after each addition. The acidity shall be calculated as oleic acid. Free Acid.

26. If soaps of iron, zinc, aluminum, or other weak bases are present, a determination of free acid is not possible, since these metallic soaps react with potassium hydroxide. Up to the present, no means has been devised whereby this determination can be made directly.

(F) Determination of Water.

27. The quantity of water shall be determined in accordance with the Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95) of the American Society for Testing Materials.[1] Water.

[1] See p. 433.

STANDARD METHOD OF TEST
FOR
BURNING QUALITY OF KEROSINE OILS[1]

Serial Designation: D 187 – 27

This method is issued under the fixed designation D 187; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1924; ADOPTED, 1927.

Scope.

1. The burning test for kerosine oil is intended for the determination of the burning quality of ordinary kerosine used for illuminating purposes.

APPARATUS[2]

Lamp.

2. *Standard Brass Saybolt Test Lamp.* (See Fig.1).—The lamp shall conform to the following requirements:

	Inches	Centimeters	Tolerances, cm.
Inside diameter of burner opening	1.21	3.08	±0.01
Top of font to top of burner opening	0.45	1.14	0.02
Outside diameter of font	5.50	13.97	....
Outside depth of font	2.49	6.33	....
Bottom of font to bottom of base	5.50	13.97	....
Filling opening			
(1) inside diameter	0.69	1.75	....
(2) distance from side of font	0.08	0.20	....
(3) height above font	0.33	0.83	....
Width of rim at bottom of font	0.65	1.65	....
Thickness of metal	0.03	0.07	....

Burner.

3. *Miller No. 2 Sun Hinge Burner* (See Fig. 2) or any other burner meeting the following:

The burner shall conform to the following requirements:

	Inches	Centimeters	Tolerances, cm.
Thickness of metal 24 B. & S. gage	....	....	....
Height of cone from base of cone (outside)	1.20	3.05	0.05
Inside diameter of cone at base	1.81	4.60	0.02

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

[2] The equipment used in this test is selected to secure results in a reasonably short time and it should be understood that this does not indicate any preference as to the proper type for general household use.

	INCHES	CENTIMETERS	TOLERANCES, CM.
Inside diameter of cone at base of flame spreader			
(1) parallel to slot	1.43	3.64	0.02
(2) perpendicular to slot	1.43	3.62	0.02
Height of cone from base to a point 1 cm.			
(1) right of center	1.15	2.92	0.06
(2) left of center	1.15	2.92	0.06
Overall diameter of flame spreader at base	2.76	7.01	0.01
Height of collar at flame spreader base	0.31	0.79	0.07
Diameter of scallops	0 21	0.54	....
Depth of scallops	0.08	0.20	....
Center of screw to bottom of collar (outside)	0.29	0.73	....
Holes at base of spreader 28 in number,			
(1) diameter	0.09	0.24	0.01
(2) spacing, edge to edge	0.14	0.35	0.02
Space at back of flame spreader collar (without holes)	0.83	2.10	....
Space at front of flame spreader collar (without holes)	0.61	1.55	....
Width of flame spreader slot at top			
(1) center	0.39	1.00	0.03
(2) 1 cm. to left of center	0.39	1.00	0.03
(3) 1 cm. to right of center	0.39	1.00	0.03
Width of wick holder (outside)	0.96	2.44	0.01
Depth of wick holder (outside)	0.17	0.44	0.01
Length of wick holder	1.75	4.45	0.02
Width of air vent (outside)	0.34	0.87	....
Depth of air vent (outside)	0.08	0.20	....
Length of air vent	1.67	4.25	0.01
Top of wick holder to base of flame spreader slot	0.03	0.08	0.06
Base of flame spreader slot (inside) to edge of wick holder (outside)	0.23	0.59	0.07
Edge of flame spreader slot (top center) to top center edge of wick holder	0.11	0.29	0.05
Wick holder (to run parallel to flame spreader slot.)			
LOWER HALF:			
Diameter at widest point (parallel to slot)	2.22	5.64	0.02
Diameter at first constriction	2.18	5.53	0.02
Diameter at second constriction	1.96	4.97	0.02
Diameter at third constriction	1.42	3.60	0.02
Diameter at fourth constriction	1.22	3.10	0.02
Diameter at bottom	1.19	3 02	0.02
Ventilating openings, 12 in number, width,			
(1) center	0.29	0.73	0.01
(2) top and bottom	0.21	0.54	0.01
Ventilating openings, height,			
(1) center	0.24	0.61	0.01
(2) sides	0.32	0.81	0.01
Top of wick holder to top of holder support	1.01	2.57	0.03
Top of lower half of burner to outside holder support	0.62	1.57	0.02

SCREEN:	INCHES	CENTIMETERS	TOLERANCES, CM.
Diameter of screen	2.12	5.38	0.02
Length of slot in screen	1.01	2.56	0.05
Width of slot (1) narrowest point	0.20	0.52	0.02
Width of slot (2) widest point	0.30	0.75	0.02
Diameter of holes in screen	0.05	0.12	0.03
Number of holes in screen per sq. cm., 45, with a tolerance of 7			
Height of screen from base to top (over-all)	0.11	0.29	0.01
Diameter of screen at top	1.16	2.94	0.02
Diameter of screen at rim	0.10	0.26	0.01

NOTE.—The burner shall be used as received from the manufacturer provided it meets the above specifications and is free from gross imperfections of manufacture. It shall be clean and in good condition at the start of the test, but emery paper shall not be used in cleaning nor imperfections removed by means of a file or otherwise

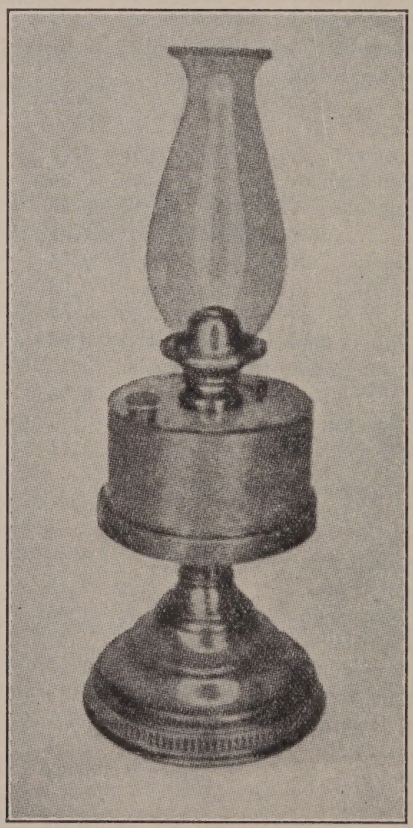


FIG 1.—Test Lamp and Chimney.

Chimney.

4. *Macbeth-Evans No. 514 Pearl Top Chimney.* (See Fig. 1.)—The chimney shall conform to the following requirements, all figures being outside measurements:

	Inches	Centimeters	Tolerances, cm.
Total height	8.06	20.48	0.50
Diameter of constriction			
(1) upper	1.77	4.50	0.08
(2) lower	2.27	5.77	0.07
Diameter at widest point	3.69	9.36	....
Diameter at top	2.83	7.20	0.09
Diameter at bottom	2.60	6.61	0.06
Distance from bottom to narrowest point	7.03	17.86	0.55
Distance from bottom to widest point	2.47	6.27	0.55
Distance from bottom to constriction near bottom	0.30	0.77	0.19
Thickness of glass at constriction			
(1) top	0.07	0.19	0.05
(2) bottom	0.05	0.12	0.03

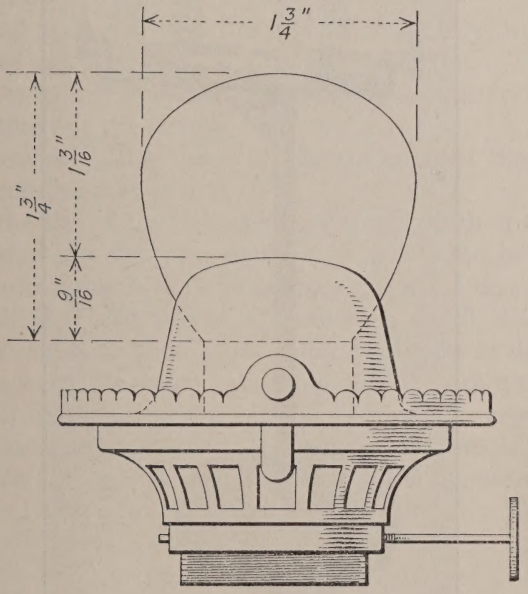


FIG. 2.—Burner.

5. *American Wick Co.'s "B" or Similar No. 2 Wick.*—The wick shall be used as received from the manufacturer and shall not be extracted with ether or other solvent previous to use. A wick shall be rejected if it fits the wick guide too loosely or too tightly, and a new wick shall be used for each burning test. **Wick.**

6. A sight gage for measuring the flame height and width accurate to $\frac{1}{16}$ in. shall be used. (See Fig. 3.) **Sight Gage.**

7. A special room for conducting the burning test is desirable but the lamp may be burned in any part of any adequately ventilated room **Room.**

reasonably free from drafts. The temperature of the room is immaterial, but it shall not vary more than 20° F. during the test.

PROCEDURE

Setting the Lamp.

8. The lamp shall be cleaned and 850 cc. of the oil to be tested shall be poured into the font. The wick, wetted with the oil, shall be inserted in the burner, and the burner shall be firmly screwed into the

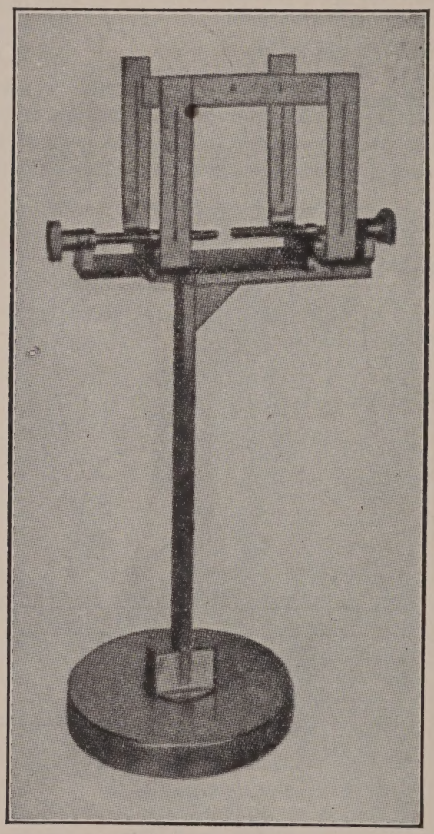


FIG. 3.—Sight Gage.

font. The wick shall be trimmed with the scissors so as to produce a flame, which, unless otherwise specified, shall be $1\frac{3}{4}$ in. in height measured from the top of the wick guide and $1\frac{3}{4}$ in. in width at its widest point. (See Fig. 2.) The trimming should be done very carefully to produce a smooth symmetrical flame free from peaks or ears, and the lamp should be lit and the flame inspected from time to time during the course of trimming in order to determine whether the trim is satisfactory. Proper height can be obtained by turning the

wick up or down and the proper width obtained by trimming the corners of the wick. These adjustments should be made to $\pm\frac{1}{16}$ in. from the standard flame dimensions. The lamp shall be allowed to burn for one hour, and the flame then readjusted, if necessary, to the standard dimensions. At the end of the first hour the lamp shall be weighed, while burning, on a platform balance accurate to 1 g., and weighed again after exactly 60 min. further burning. The oil consumed shall be calculated in cubic centimeters per hour. The initial rate of oil consumption, thus established, shall not vary from 43 cc. per hour by more than ±2 cc. With proper attention to the flame dimensions this measurement is needed only as a check, since with a given flame size and shape the initial rate of oil consumption will always be within the limit set.

Conducting the Test.

9. The oil shall be allowed to burn continuously, without further adjustment of any kind, for the duration of the test. The lamp shall be refilled to within $\frac{1}{4}$ in. of the top of the fount, at intervals of 8 hours, without putting out the flame.

Duration of the Test.

10. Unless otherwise specified, the duration of the test shall be 24 hours of continuous burning.

Interpretation.

11. At the end of the test, the height and width of the flame and the condition of the chimney, flame, and wick, shall be noted. The final flame measurement shall be taken without refilling the lamp.

Reproducibility.

12. With care and proper attention to detail, the average oil consumption per hour should not differ in duplicate determinations by more than 2 cc. With this condition fixed, the other characteristics of the oil will be reproduced invariably, with the exception of initial mushroom formations ("ears," "niggers," "toadstools"), reproducibility of which will vary with the quality of the oil, amounting in the poorer oils to 10 per cent of the burning time.

STANDARD METHOD OF TEST
FOR
BURNING QUALITY OF LONG-TIME BURNING OIL FOR RAILWAY USE[1]

Serial Designation: D 219 – 27

This method is issued under the fixed designation D 219; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1925; ADOPTED IN AMENDED FORM, 1927.

Scope.

1. The burning test for long-time burning oil is intended for the determination of the burning quality of special kerosine oil used in railway semaphore signal lamps.

APPARATUS

Lamp.

2. The lamp shall be the standard American Railway Association, Signal Section, semaphore lamp.

Font.

3. A 31 fluid ounce font, about 3 in. high, measured from inside the bottom to top of burner collar, and fitting the standard lamp, shall be used.

Burner.

4. The burner shall be a *Dressel No. 23 Long-Time Burner*, New York Central Type, or any other burner meeting the following requirements: A nickel plated long-time burner made of suitable weight metal, provided with the conventional wick adjusting device, with any convenient means of fitting to font, and in other respects corresponding to the following requirements:

Flame Spreader.—Cylindrical, $\frac{5}{16}$ in. in height and $\frac{1}{2}$ in. in inside diameter, soldered to the top of wick tube. Two V-shaped slots, placed opposite to each other, with top of V $\frac{1}{4}$ in. wide and bottom ending just above top of wick tube.

Wick Tube.—$1\frac{5}{16}$ in. in length, $\frac{1}{4}$ in. in inside diameter, and extending $\frac{5}{8}$ in. above top of wick tube.

Chimney Support Ring.—$1\frac{1}{4}$ in. in inside diameter, $\frac{7}{16}$ in. below top of flame spreader, equipped with 4 springs to hold chimney in place.

Air Vents.—Four $\frac{1}{16}$ in. holes placed symmetrically in flame spreader just above the top of the wick tube; four rectangular

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

openings, $\frac{7}{16}$ in. high, by $\frac{9}{16}$ in. wide, placed below the chimney support ring and between the chimney springs.

5. The chimney shall be a No. 55-D Chimney,[1] or any other chimney meeting the following requirements: Straight wall glass $2\frac{1}{2}$ in. high and $1\frac{1}{8}$ in. in inside diameter, with rim at top and bottom. **Chimney.**

6. A new gray round felt wick, approximately $7\frac{3}{4}$ in. long and $\frac{3}{16}$ in. in diameter, made by sewing a strip of felt $\frac{1}{2}$ in. wide by $7\frac{3}{4}$ in. long by $\frac{1}{8}$ in. thick into proper form is suitable. The wick shall be washed with redistilled ether and dried at room temperature before use. **Wick.**

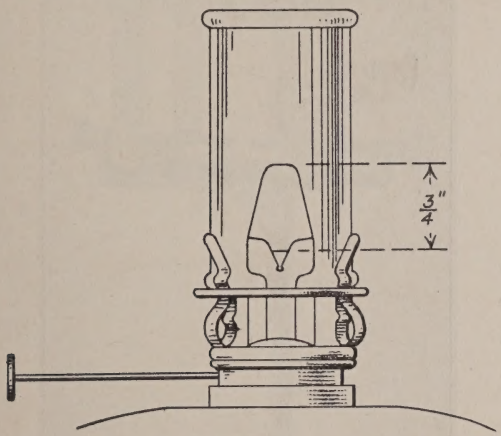


FIG 1

7 A platform balance sensitive to 1 g. shall be used. **Balance.**

8. The lamp may be burned in any well ventilated room, or out of doors, if not subjected to the direct rays of the sun. **Room.**

9. A sight gage, or other suitable flame measuring device, accurate to $\frac{1}{16}$ in. shall be used. The sight gage shown in Fig. 2 is satisfactory. **Sight Gage.**

PROCEDURE

10. Seven hundred and fifty cubic centimeters of the oil to be tested shall be placed in the font. The burner, wick, chimney and font shall be assembled, the rough edge trimmed from the wick and the wick centered in the flame spreader, lighted and adjusted to give a flame $\frac{3}{4}$ in. high, measured from top of wick. This, and all following flame measurements, should be made with the assembly inside of the lamp, with slide open, and in a room free from drafts. **Procedure.**

[1] This term, "55-D Chimney," is fully descriptive with glass manufacturing companies as the long-time burner chimney covered by these specifications.

The flame should have a flat top and be of the general shape indicated in Fig. 1. If this cannot be accomplished by leveling the lamp, the burner should be rejected. Inability to secure the specified flame shape will be due, in such cases, to improper alignment of the sides of the flame spreader. It is essential that the specified flame shape be adhered to, as any deviation from it may influence the rate of oil

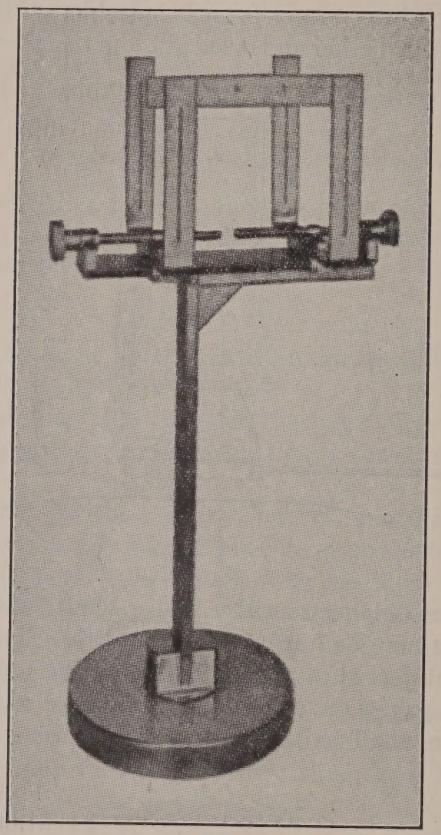


FIG. 2.—Sight Gage.

consumption and the flame drop. The assembly shall be weighed while burning, then placed in the lamp. The wick shall be readjusted, if necessary, without removing the font from the lamp, during the first four hours. The lamp shall be allowed to burn continuously for the duration of the test. After 96 hours, the font assembly shall be removed from the lamp, weighed while burning and then replaced in the lamp. After 120 hours the lamp shall again be weighed and the

average rate of oil consumption per hour calculated for the 96 to 120-hour period. From this rate and the original weight of oil in the font, the number of additional hours required to make the total quantity of oil consumed 650 cc. shall be calculated and the lamp burned the calculated length of time. The oil consumption shall be verified by weighing at the end of the test. The time required to burn 650 cc. will, in general, be not less than 120 hours and not more than 144 hours if proper attention is paid to initial flame height and shape. At the end of the test, observe and record the change in height of flame, and condition of the wick and chimney.

STANDARD METHOD OF TEST

FOR

BURNING QUALITY OF MINERAL SEAL OIL[1]

Serial Designation: D 239 – 27

This method is issued under the fixed designation D 239; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1926; ADOPTED, 1927.

Scope. 1. The burning test for mineral colza oil is intended for the determination of the burning quality of the special illuminating oil known to the trade as mineral seal oil, 300 oil or mineral colza oil, used in railway coach lamps.

APPARATUS

Lamp. 2. The lamp shall be the standard Dressel No. 520 Side Lamp fitted with a No. 3 Dual Burner without spring chimney holder and without smoke bell.

Font. 3. A 24-fluid-oz. font having the following measurements shall be used:

	Inches	Centimeters	Tolerances, cm.
Inside diameter of burner opening	1.39	3.52	±0.01
Outside diameter of font at bottom	3.74	9.49	....
Outside diameter of font above constriction	4.06	10.32	....
Outside diameter of font at widest point	4.50	11.43	....
Outside diameter of font at neck	1.85	4.69	....
Height of font (to top of collar)	4.31	10.95	....
Height of font (to outside edge top)	3.65	9.27	0.08
Diameter of inside compartment	1.89	4.80	....
Thickness of metal	0.03	0.07	....

Burner. 4. The burner shall be a Dressel "D" or No. 3 Dual Burner, without skirt, or any other burner meeting the following requirements:

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

	Inches	Centimeters	Tolerances cm.
UPPER PART OF BURNER:			
Thickness of metal, No. 24 B. & S. gage.			
Height of cone from base of cone:			
(1) Center	1.61	4.09	0.03
(2) 1 cm. to right of center	1.57	3.98	0.03
(3) 1 cm. to left of center	1.56	3.97	0.05
Inside diameter of cone at base	2.05	5.20	....
Inside diameter of cone at constriction	2.00	5.08	....
Inside diameter of cone at base of flame spreader slot:			
(1) Parallel to slot	1.49	3.79	0.01
(2) Perpendicular to slot	1.52	3.85	0.01
Height of collar	1.95	4.96	....
Holes near base of cone, 18 in number:			
(1) Diameter	0.11	0.27	....
(2) Spacing edge to edge	0.13	0.33	....
(3) Spaces at sides of cone (without holes)	0.78	1.98	....
Width of flame spreader slot:			
(1) Center	0.31	0.78	0.02
(2) 1 cm. to right of center	0.31	0.78	0.02
(3) 1 cm. to left of center	0.31	0.78	0.02
Ventilating openings, 40 in number in each row:			
(1) Top row (diamond shaped), height and width	0.13	0.33	....
(2) Second row (circular shaped) diameter	0.09	0.22	....
(3) Third row (diamond shaped) height and width	0.13	0.33	....
(4) Fourth row (circular shaped) diameter	0.09	0.22	....
(5) Bottom row (diamond shaped):			
Height	0.22	0.55	....
Width	0.11	0.28	....
LOWER PART OF BURNER:			
Diameter of widest point	2.31	5.87	....
Diameter at narrowest point (just below screen)	1.28	3.26	....
Diameter at bottom (thread)	1.40	3.55	....
Top of screen (outer edge) to bottom of burner	1.65	4.19	0.06
Top of wick holder to top of screen	0.91	2.30	0.08
Holes in screen:			
(1) Outer circle, 36 in number, diameter	0.11	0.27	....
(2) Second circle, 36 in number, diameter	0.11	0.27	....
(3) Inner circle, 36 in number, diameter	0.07	0.17	....
Space between wick holders	0.11	0.27	0.04
Wick holders:			
(1) Width (outside)	1.18	3.00	....
(2) Depth (outside)	0.19	0.48	0.06
(3) Length	2.54	6.44	0.02
Wick holder brace:			
(1) Width (outside)	1.02	2.60	....
(2) Depth (outside)	0.15	0.38	....

FOLLOWING MEASUREMENTS TAKEN WITH UPPER PART OF BURNER FITTED EVENLY AND TIGHTLY ON LOWER HALF:

	INCHES	CENTIMETERS	TOLERANCES, CM.
Top of wick holder to base of flame spreader slot	0.32	0.81	0.03
Base of flame spreader slot (inside) to edge of wick holder (outside)	0.13	0.34	0.04
Height of burner (overall)	3.24	8.23	0.03

Chimney. 5. The chimney shall be a Keystone No. 24 Chimney, or any other chimney meeting the following requirements:[1]

	INCHES	CENTIMETERS	TOLERANCES, CM.
Total height	9.94	25.24	....
Diameter at top	1.60	4.06	0.02
Diameter at a point 1.75 in. (4.44 cm.) from top	1.60	4.06	0.02
Diameter at a point 5 in. from top	2.60	6.60	....
Diameter at widest point	3.58	9.10	....
Diameter at the bottom	2.55	6.47	0.03
Distance from bottom to widest point	2.69	6.78	0.11
Distance from bottom to constriction near bottom	0.94	2.38	....
Thickness of glass at top	0.06	0.14	0.01
Thickness of glass at bottom	0.05	0.12	0.03

Wick. 6. The wicks shall be the American "D" Dual Wick and shall be used as received from the manufacturer. New wicks shall be used in each test.

Balance. 7. A platform balance sensitive to 0.1 g. shall be used.

Room. 8. The lamp shall be burned in a well-ventilated room, free from draughts, and not subjected to the direct rays of the sun. The lamp shall be mounted on a wall, at a distance of approximately $2\frac{1}{4}$ in. from the wall, by means of a bracket provided with the lamp.

Sight Gage. 9. A sight gage, or some other suitable flame measuring device, accurate to $\frac{1}{16}$ in. shall be used. The sight gage shown in Fig. 1 is satisfactory.

PROCEDURE

Setting the Lamp. 10. Place approximately 600 cc. of the oil to be tested in a clean font. The wicks, wetted with the oil, shall be inserted in the burner and then assembled with the font and chimney. The wicks shall then be trimmed with scissors so that they are of the same height and so as to produce a flame which, unless otherwise specified, shall be $1\frac{1}{16}$ in. high measured from the top of the wick guide, and $1\frac{3}{8}$ in. wide at its widest point. The trimming shall be done very carefully to produce a flame free from peaks or ears and the lamp shall be lit and the flame frequently inspected during the course of the trimming in order to determine the conditions of the flame as to size and shape. Proper

[1] All measurements are outside.

height can be obtained by turning up or down, and the proper width by trimming the corners of the wick. These adjustments shall be made to $\pm\frac{1}{16}$ in. from the standard flame. After the proper flame has been obtained, the burner and wicks shall be removed, the wicks dried as thoroughly as possible without removing the wicks from the burner and the apparatus assembled with a clean font and chimney.

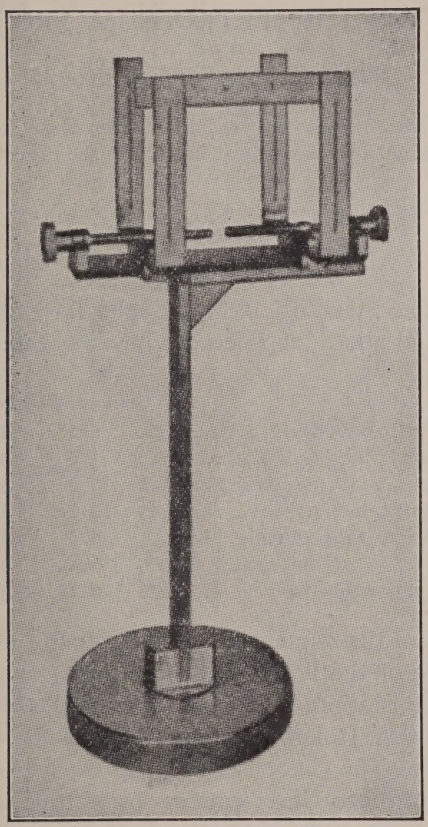


FIG. 1.—Sight Gage.

Weigh the assembly and then pour exactly 600 cc. of the sample into the font, then light the lamp and weigh again while burning. After 1 hour, the flame shall be measured and adjusted if necessary to the proper height and width. Allow the lamp to burn continuously until 570 cc. of oil have been consumed, as determined by weight. In general, 570 cc. of oil will be consumed in not less than 20 hours. At the end of the test, record the number of hours burned, the flame change and the condition of wick and chimney.

STANDARD METHOD OF TEST
FOR
DISTILLATION OF GASOLINE, NAPHTHA, KEROSINE, AND SIMILAR PETROLEUM PRODUCTS[1]

Serial Designation: D 86 – 27

This method is issued under the fixed designation D 86; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1927.

APPARATUS

Flask. 1. *Flask.*—The Standard 100-cc. Engler flask is shown in Fig. 1, the dimensions and allowable tolerance being as follows:

	Centimeters	Inches	Tolerances, cm.
Diameter of bulb, outside	6.5	2.56	0.2
" " neck, inside	1.6	0.63	0.1
Length of neck	15.0	5.91	0.4
" " vapor tube	10.0	3.94	0.3
Diameter of vapor tube, outside	0.6	0.24	0.05
" " " " inside	0.4	0.16	0.05
Thickness of vapor tube wall	0.1	0.04	0.05

The position of the vapor tube shall be 9 cm. (3.55 in.) ±3 mm. above the surface of the liquid when the flask contains its charge of 100 cc. The tube is approximately in the middle of the neck and set at an angle of 75 deg. (tolerance ±3 deg.) with the vertical.

Condenser. 2. *Condenser.*—The condenser (Fig. 2) consists of a $\frac{9}{16}$-in. (14.29 mm.) OD No. 20 Stubbs Gage seamless brass tube, 22 in. (55.88 cm.) long. It is set at an angle of 75 deg. from the perpendicular and is surrounded with a cooling bath 15 in. long (38.1 cm.), approximately 4 in. (10.16 cm.) wide by 6 in. (15.24 cm.) high. The lower end of the condenser tube is cut off at an acute angle, and curved downward for a length of 3 in. (7.62 cm.) and slightly backward so as to insure contact with the wall of the graduate at a point 1 to $1\frac{1}{4}$ in. (2.54 to 3.8 cm.) below the top of the graduate when it is in position to receive the distillate.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

3. *Shield.*—The shield (Fig. 2) is made of approximately 22 gage sheet metal and is 19 in. (48.26 cm.) high, 11 in. (27.94 cm.) long and 8 in. (20.32 cm.) wide, with a door on one narrow side, with two openings, 1 in. (2.54 cm.) in diameter, equally spaced, in each of Shield.

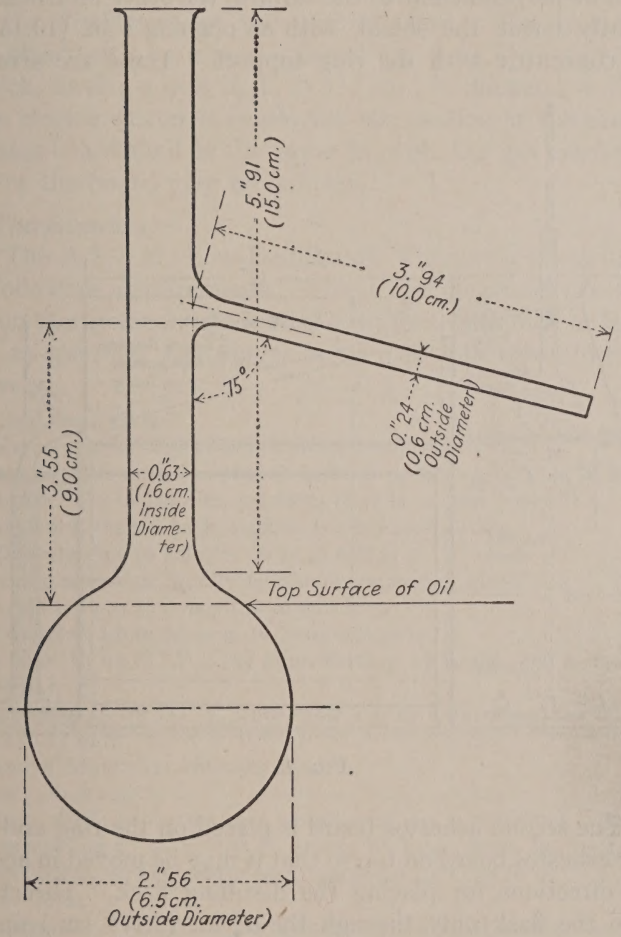


FIG. 1.

the two narrow sides, and with a slot cut in one side for the vapor tube. The centers of these four openings are $8\frac{1}{2}$ in. (21.59 cm.) below the top of the shield. There are also three $\frac{1}{2}$-in. (1.27 cm.) holes in each of the four sides with their centers 1 in. (2.54 cm.) above the base of the shield.

Support.

4. *Ring Support and Hard Asbestos Boards.*—The ring support is of the ordinary laboratory type, 4 in. (10.16 cm.) or larger in diameter, and is supported on a stand inside the shield. There are two hard asbestos boards: One 6 by 6 by $\frac{1}{4}$ in. (15.24 by 15.24 cm. by 6.35 mm.) with a hole $1\frac{1}{4}$ in. (3.175 cm.)[1] in diameter in its center, the sides of which shall be perpendicular to the surface; the other, an asbestos board to fit tightly inside the shield, with an opening 4 in. (10.16 cm.) in diameter concentric with the ring support. These are arranged as

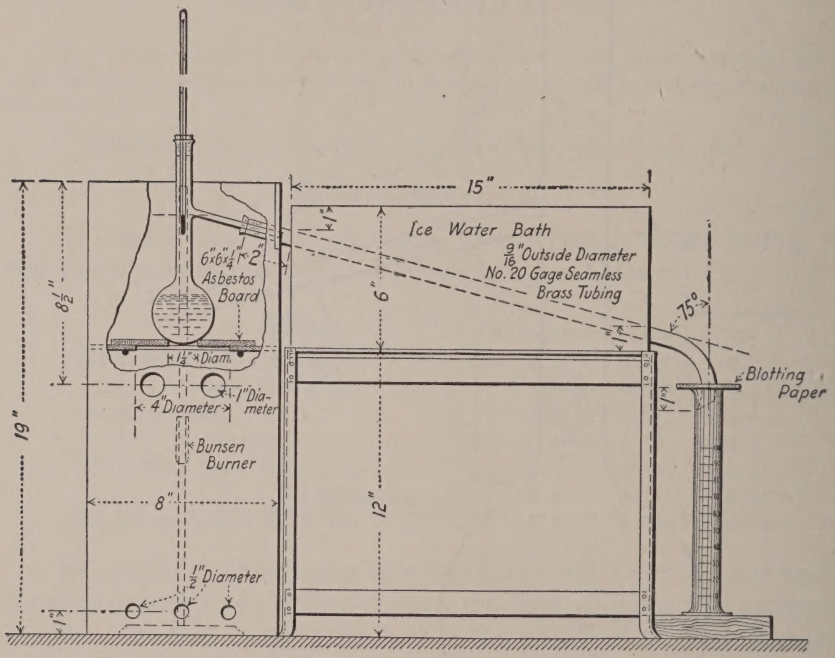


FIG. 2.

follows: The second asbestos board is placed on the ring and the first or smaller asbestos board on top so that it may be moved in accordance with the directions for placing the distilling flask. Direct heat is applied to the flask only through the $1\frac{1}{4}$-in. (3.175 cm.) opening in the first asbestos board.

Heater

5. *Gas Burner or Electric Heater.*—

(*a*) *Gas Burner.*—The burner is so constructed that sufficient heat can be obtained to distill the product at the uniform rate specified below. The flame should never be so large that it spreads over a

[1] When distilling petroleum products having an end point above 470° F. (243.34° C.), the hole in the asbestos board shall be $1\frac{1}{2}$ in. (3.81 cm.) in diameter.

circle of diameter greater than $3\frac{1}{2}$ in. (8.89 cm.) on the under surface of the asbestos board. A sensitive regulating valve is a necessary adjunct, as it gives complete control of heating.

(*b*) *Electric Heater.*—The electric heater, which may be used in place of the gas flame, shall be capable of bringing over the first drop within the time specified below when started cold, and of continuing the distillation at the uniform rate. The electric heater shall be fitted with an asbestos board top $\frac{1}{8}$ to $\frac{1}{4}$ in. (3.175 to 6.35 mm.) thick, having a hole $1\frac{1}{4}$ in. (3.175 cm.) in diameter in the center. When an electric heater is employed, the portion of the shield above the asbestos board shall be the same as with the gas burner but the part below the board may be omitted.

6. *Thermometers.*— Thermometers.

(*a*) The A.S.T.M. Low-Distillation Thermometer shall conform to the following requirements. These specifications cover a total-immersion thermometer graduated in either Centigrade or Fahrenheit degrees, as specified, the ranges being 0 to 300° C. or 30 to 580° F., respectively.

TYPE: Etched stem, glass.

LIQUID: Mercury.

RANGE AND SUBDIVISION: 0 to 300° C. in 1° C. or 30 to 580° F. in 2° F.

TOTAL LENGTH: 378 to 384 mm. (14.88 to 15.12 in.).

STEM: Plain front, enamel back, suitable thermometer tubing.
Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).

BULB: Corning normal or equally suitable thermometric glass.
Length, 10 to 15 mm. (0.39 to 0.59 in.).
Diameter, 5.0 to 6.0 mm. (0.20 to 0.24 in.).

DISTANCE TO 0° C. OR 32° F. LINE FROM BOTTOM OF BULB: 100 to 110 mm. (3.94 to 4.33 in.).

DISTANCE TO 300° C. OR 572° F. LINE FROM TOP OF THERMOMETER: 30 to 45 mm. (1.18 to 1.77 in.).

FILLING ABOVE MERCURY: Nitrogen gas.

TOP FINISH: Glass ring.

GRADUATION: All lines, figures, and letters clear cut and distinct. The first and each succeeding 5° C. or 10° F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.

IMMERSION: Total.

SPECIAL MARKING: "A.S.T.M. Low Distillation," a serial number and the manufacturer's name or trade mark shall be etched on the stem.

SCALE ERROR: The error at any point of the scale when the thermometer is standardized as provided below shall not exceed 0.5° C. or 1° F.

STANDARDIZATION: The thermometer shall be standardized immersed in the testing bath to the top of the mercury column, at the ice point and at temperature intervals of approximately 50° C. or 100° F. up to 300° C. or 572° F.

TEST FOR PERMANENCY OF RANGE: After being subjected to a temperature of 280 to 290° C. or 540 to 560° F. for 24 hours, the accuracy shall be within the limit specified.

CASE: The thermometer shall be supplied in a suitable case on which shall appear The marking: "A.S.T.M. Low Distillation, 0 to 300° C." or "A.S.T.M. Low Distillation, 30 to 580° F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

(*b*) The A.S.T.M. High-Distillation Thermometer shall conform to the following requirements. These specifications cover a total-immersion thermometer graduated either in Centigrade or Fahrenheit degrees, as specified, the ranges being 0 to 400° C. or 30 to 760° F., respectively.

TYPE: Etched stem, glass.

LIQUID: Mercury.

RANGE AND SUBDIVISION: 0 to 400° C. in 1° C. or 30 to 760° F. in 2° F.

TOTAL LENGTH: 378 to 384 mm. (14.88 to 15.12 in.).

STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).

BULB: Corning normal or equally suitable thermometric glass.

Length, 10 to 15 mm. (0.39 to 0.59 in.).

Diameter, 5.0 to 6.0 mm. (0.20 to 0.24 in.).

DISTANCE TO 0° C. OR 32° F. LINE FROM BOTTOM OF BULB: 25 to 35 mm. (0.98 to 1.38 in.).

DISTANCE TO 400° C. OR 752° F. LINE FROM TOP OF THERMOMETER: 30 to 45 mm. (1.18 to 1.77 in.).

FILLING ABOVE MERCURY: Nitrogen gas.

TOP FINISH: Glass ring.

GRADUATION: All lines, figures and letters clear cut and distinct. The first and each succeeding 5° C. or 10° F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.

IMMERSION: Total.

SPECIAL MARKING: "A.S.T.M. High Distillation" a serial number and the manufacturer's name or trade mark shall be etched on the stem.

SCALE ERROR: The error at any point of the scale up to 370° C. or 700° F. when the thermometer is standardized as provided below shall not exceed 1° C. or 2° F.

STANDARDIZATION: The thermometer shall be standardized immersed in the testing bath to the top of the mercury column, at the ice point and at temperature intervals of approximately 50° C. or 100° F. up to 370° C. or 700° F.

TEST FOR PERMANENCY OF RANGE: After being subjected to a temperature between 360 and 370° C. or 680 and 700° F. for 24 hours, the accuracy shall be within the limit specified.

CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. High Distillation, 0 to 400° C." or "A.S.T.M. High Distillation, 30 to 760° F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

7. *Graduate.*—The graduate shall be of the cylindrical type, of uniform diameter, with a pressed or molded base and a lipped top. The cylinder shall be graduated to contain 100 cc., and the graduated portion shall be not less than 7 in. (17.78 cm.) nor more than 8 in. (20.32 cm.) long. It shall be graduated in single cubic centimeters and each fifth mark shall be distinguished by a longer line. It shall be numbered from the bottom up at intervals of 10 cc. The overall height of the graduate shall be not less than $9\frac{3}{4}$ in. (24.8 cm.), nor more than $10\frac{1}{4}$ in. (26.0 cm.). The graduations shall not be in error by more than 1 cc. at any point on the scale. Graduate.

PROCEDURE

8. (*a*) The condenser bath shall be filled with cracked ice,[1] and enough water added to cover the condenser tube. The temperature shall be maintained between 32 and 40° F. (0 and 4.45° C.). Procedure.

(*b*) The condenser tube shall be swabbed to remove any liquid remaining from the previous test. A piece of soft cloth attached to a cord or copper wire may be used for this purpose.

(*c*) One hundred cubic centimeters of the product shall be measured in the 100-cc. graduated cylinder at 55 to 65° F. (12.78 to 18.33° C.) and transferred directly to the Engler flask. None of the liquid shall be permitted to flow into the vapor tube.

(*d*) The thermometer[2] provided with a cork shall be fitted tightly into the flask so that it will be in the middle of the neck and so that the lower end of the capillary tube is on a level with the inside of the bottom of the vapor outlet tube at its junction with the neck of the flask. The thermometer shall be approximately at room temperature when placed in the flask.

(*e*) The charged flask shall be placed in the $1\frac{1}{4}$-in. (3.175-cm.) opening in the 6 by 6-in. (15.24 by 15.24-cm.) asbestos board with the vapor outlet tube inserted into the condenser tube. A tight connection may be made by means of a cork through which the vapor tube passes. The position of the flask shall be so adjusted that the

[1] Any other convenient cooling medium may be used.

[2] For products having an initial boiling point of 212° F. (100° C.) or higher, the High-Distillation Thermometer shall be used; for all other products, the Low-Distillation Thermometer shall be used.

vapor tube extends into the condenser tube not less than 1 in. (2.54 cm.) nor more than 2 in. (5.08 cm.).

(*f*) The graduated cylinder used in measuring the charge shall be placed, without drying, at the outlet of the condenser tube in such a position that the condenser tube shall extend into the graduate at least 1 in. (2.54 cm.) but not below the 100-cc. mark. Unless the temperature is between 55 and 65° F. (12.78 and 18.33° C.) the receiving graduate shall be immersed up to the 100-cc. mark in a transparent bath maintained between these temperatures. The top of the graduate shall be covered closely during the distillation with a piece of blotting paper or its equivalent, cut so as to fit the condenser tube tightly.

Distillation.

9. When everything is in readiness, heat shall be applied at a uniform rate, so regulated that the first drop of condensate falls from the condenser in not less than 5 nor more than 10 minutes. The distillation thermometer shall be read 2 minutes after heat is applied and the indication recorded as the "correction temperature." This figure is of significance only in cases when there is a question as to the accuracy[1] of the initial boiling point, as subsequently determined. When the first drop falls from the end of the condenser the reading of the distillation thermometer shall be recorded as the *initial boiling point.* The receiving cylinder shall then be moved so that the end of the condenser tube shall touch the side of the cylinder. The heat shall then be so regulated that the distillation will proceed at a uniform rate of not less than 4 nor more than 5 cc. per minute. The volume of distillate collected in the cylinder shall be observed and recorded, to the nearest 0.5 cc., when the mercury of the thermometer reaches each point that is an even multiple of 25° C. or the Fahrenheit equivalent of this point (50° C., 75° C., 100° C., 125° C., etc., or 122° F., 167° F., 212° F., 257° F., etc.). If preferred, the reading of

[1] The initial boiling point is one of the observations which it is customary to report as indicative of the quality of gasoline. It is a point which is very difficult to check, especially when duplicate tests are made at different room temperatures, and it has not been found practical to devise an accurate method of correcting for this unavoidable variation in operating conditions. The practical significance of initial boiling point is not regarded as sufficient to warrant requiring that tests be conducted under regulated conditions of room temperature. The following directions are included as an expedient intended to obviate some of the difficulties that develop on account of the practice of including initial boiling point limits in specifications for the purchase and sale of gasoline:

If the "correction temperature" is below 70° F. (21° C.) or above 80° F. (27° C.), the observed initial boiling point, if it is below 150° F. (66° C.), may be considered incorrect by an amount not less than one third the difference between the "correction temperature" and 75° F. (24° C.). In case there is a dispute between buyer and seller, the observed initial boiling point figures obtained by all parties shall be revised according to the following formula:

$$\text{Revised initial boiling point} = \left\{\begin{matrix}\text{observed initial}\\ \text{boiling point}\end{matrix}\right\} - \frac{\text{Correction Temperature (deg. Fahr.)} - 75}{3}$$

If all of the revised figures fall within the specification limit the gasoline in question shall be considered as passing; otherwise arrangements shall, if possible, be made to conduct a distillation test with the room temperature maintained between the limits of 70 and 80° F. (21 and 27° C.) inclusive.

the distillation thermometer may be observed and recorded when the level of the distillate reaches each 10-cc. mark on the graduate. In case a product is being tested to ascertain whether or not it conforms with a given specification, all necessary observations shall be made and recorded, whether or not they are included in the series ordinarily employed by the laboratory making the test.

When the liquid residue in the distillation flask is approximately 5 cc. the heat may be increased because of the presence of heavy ends which have relatively high boiling points. However, no further increase of heat shall be applied after this adjustment. The 4 to 5-cc. rate can rarely be maintained from this point to the end of the distillation, but in no case shall the period between the point when approximately 5 cc. of liquid remains in the flask and the end point be more than 5 minutes.

The heating shall be continued until the mercury reaches a maximum and starts to fall consistently. The highest temperature observed on the distillation thermometer shall be recorded as the *maximum temperature* or end point. Usually this point will be reached after the bottom of the flask has become dry.

The total volume of the distillate collected in the receiving graduate shall be recorded as the *recovery*.

The cooled residue shall be poured from the flask into a small cylinder graduated in 0.1 cc., measured when cool and the volume recorded as *residue*.

The difference between 100 cc. and the sum of the recovery and the residue shall be calculated and recorded as *distillation loss*.

ACCURACY

Accuracy. 10. With proper care and attention to detail, duplicate results obtained for initial boiling point and maximum temperature, respectively, should not differ from each other by more than 6° F. (3.33° C.). Duplicate readings of the volume of distillate collected in the cylinder when each of the prescribed temperature points is reached should not differ from each other by more than 2 cc. In case observations are made on the basis of prescribed percentage points, the differences in temperature readings should not exceed the amounts equivalent to 2 cc. of distillate at each point in question.

CORRECTION FOR BAROMETRIC PRESSURE

Correction. 11. The actual barometric pressure shall be ascertained and recorded, but no correction shall be made except in case of dispute.

In such cases the temperature points shall be corrected to 760 mm. (29.92 in.), by the use of the Sydney Young equation, as follows:

For Centigrade readings:

$$C_c = 0.00012\ (760-P)\ (273 + t_c)$$

For Fahrenheit readings:

$$C_f = 0.00012\ (760-P)\ (460 + t_f)$$

in which C_c and C_f are, respectively, corrections to be added to the observed temperature t_c or t_f, and P is the actual barometric pressure in millimeters of mercury.

The following table is a convenient approximation of the corrections as calculated by the above equation.

Temperature Range		Correction[1] per 10-mm. Difference in Pressure,	
deg. Cent.	deg. Fahr.	deg. Cent.	deg. Fahr.
10 – 30	50 – 86	0.35	0.63
30 – 50	86 – 122	0.38	0.68
50 – 70	122 – 158	0.40	0.72
70 – 90	158 – 194	0.42	0.76
90 – 110	194 – 230	0.45	0.81
110 – 130	230 – 266	0.47	0.85
130 – 150	266 – 302	0.50	0.89
150 – 170	302 – 338	0.52	0.94
170 – 190	338 – 374	0.54	0.98
190 – 210	374 – 410	0.57	1.02
210 – 230	410 – 446	0.59	1.06
230 – 250	446 – 482	0.62	1.11
250 – 270	482 – 518	0.64	1.15
270 – 290	518 – 554	0.66	1.19
290 – 310	554 – 590	0.69	1.24
310 – 330	590 – 626	0.71	1.28
330 – 350	626 – 662	0.74	1.32
350 – 370	662 – 698	0.76	1.37
370 – 390	698 – 734	0.78	1.41
390 – 410	734 – 770	0.81	1.45

[1] To be added in case barometric pressure is below 760 mm.; to be subtracted in case barometric pressure is above 760 mm.

STANDARD METHOD OF TEST
FOR
DISTILLATION OF NATURAL GAS GASOLINE[1]

Serial Designation: D 216 – 27

This method is issued under the fixed designation D 216; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1925; ADOPTED, 1927.

APPARATUS

1. *Flask.*—The standard 100-cc. Engler flask is shown in Fig. 1, the dimensions and allowable tolerance being as follows: **Flask.**

	Centimeters	Inches	Tolerances, cm.
Diameter of bulb, outside	6.5	2.56	0.2
Diameter of neck, inside	1.6	0.63	0.1
Length of neck	15.0	5.91	0.4
Length of vapor tube	10.0	3.94	0.3
Diameter of vapor tube, outside	0.6	0.24	0.05
Diameter of vapor tube, inside	0.4	0.16	0.05
Thickness of vapor tube wall	0.1	0.04	0.05

The position of the vapor tube shall be 9 cm. (3.55 in.) ±3 mm. above the surface of the liquid when the flask contains its charge of 100 cc. The tube is approximately in the middle of the neck and set at an angle of 75 deg. (tolerance ±3 deg.) with the vertical.

2. *Condenser.*—The condenser (Fig. 2) consists of a $\frac{9}{16}$-in. (14.29- mm.) OD No. 20 Stubbs Gage seamless brass tube, 22 in. (55.88 cm.) long. It is set at an angle of 75 deg. from the perpendicular and is surrounded with a cooling bath 15 in. long (38.1 cm.) approximately 4 in. (10.16 cm.) wide by 6 in. (15.24 cm.) high. The lower end of the condenser tube is cut off at an acute angle, and curved downward for a length of 3 in. (7.62 cm.) and slightly backward so as to insure contact with the wall of the graduate at a point 1 to $1\frac{1}{4}$ in. (2.54 to 3.8 cm.) below the top of the graduate when it is in position to receive the distillate. **Condenser.**

3. *Shield.*—The shield (Fig. 2) is made of approximately No. 22 gage sheet metal and is 19 in. (48.26 cm.) high, 11 in. (27.94 cm.) long **Shield.**

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

and 8 in. (20.32 cm.) wide, with a door on one narrow side, with two openings, 1 in. (2.54 cm.) in diameter equally spaced, in each of the two narrow sides, and with a slot cut in one side for the vapor tube. The centers of these four openings are $8\frac{1}{2}$ in. (21.59 cm.) below the top of the shield. There are also three $\frac{1}{2}$-in. (1.27-cm.) holes in each

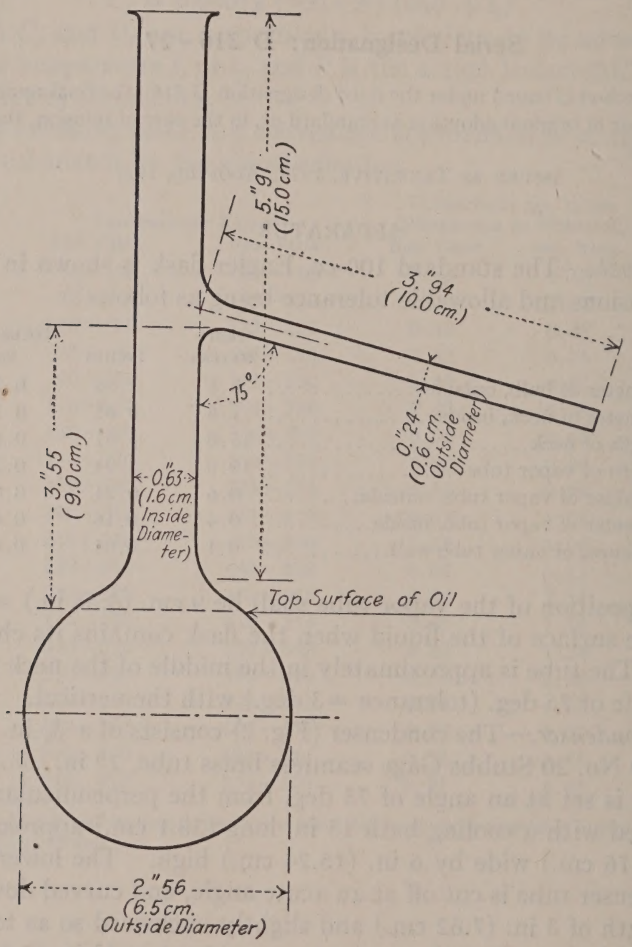


FIG. 1.

of the four sides with their centers 1 in. (2.54 cm.) above the base of the shield.

Support. 4. *Ring Support and Hard Asbestos Boards.*—The ring support is of the ordinary laboratory type, 4 in. (10.16 cm.) or larger in diameter, and is supported on a stand inside the shield. There are two hard

asbestos boards: one 6 by 6 by $\frac{1}{4}$ in. (15.24 by 15.24 cm. by 6.35 mm.), with a hole $1\frac{1}{4}$ in. (3.175 cm.) in diameter in its center, the sides of which shall be perpendicular to the surface; the other, to fit tightly inside the shield, with an opening 4 in. (10.16 cm.) in diameter concentric with the ring support. These are arranged as follows: The second asbestos board is placed on the ring, and the first or smaller asbestos board on top so that it may be moved in accordance with the directions for placing the distilling flask. Direct heat is applied to

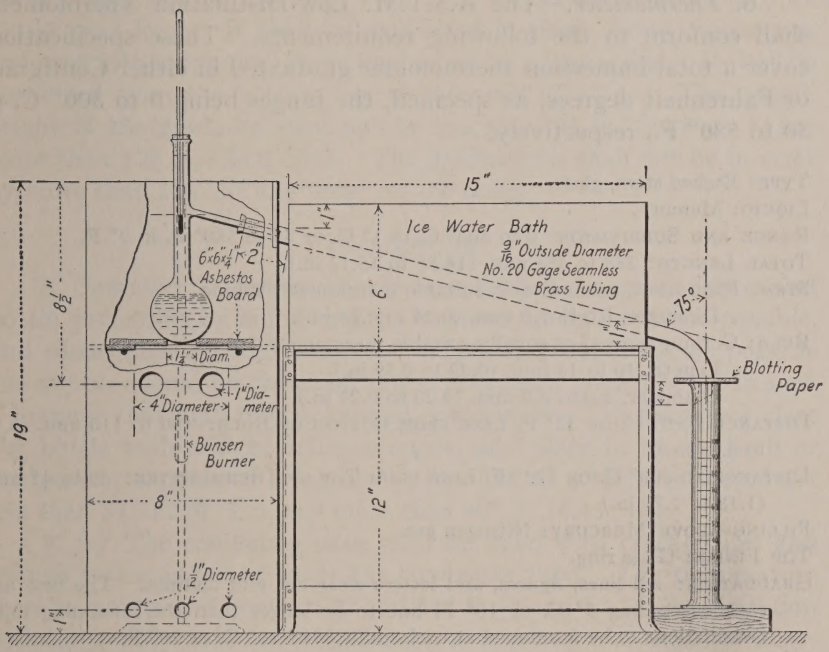


FIG. 2.

the flask only through the $1\frac{1}{4}$ in. (3.175 cm.) opening in the first asbestos board.

Heater.

5. *Gas Burner or Electric Heater.*

(*a*) *Gas Burner.*—The burner is so constructed that sufficient heat can be obtained to distill the product at the uniform rate specified below. The flame should never be so large that it spreads over a circle of a diameter greater than $3\frac{1}{2}$ in. (8.89 cm.) on the under surface of the asbestos board. A sensitive regulating valve is a necessary adjunct, as it gives complete control of heating.

(*b*) *Electric Heater.*—The electric heater, which may be used in place of the gas flame, shall be capable of bringing over the first drop within the time specified below when started cold, and of continuing the distillation at the uniform rate. The electric heater shall be fitted with an asbestos board top $\frac{1}{8}$ to $\frac{1}{4}$ in. (3.175 to 6.35 mm.) thick, having a hole $1\frac{1}{4}$ in. (3.175 cm.) in diameter in the center. When an electric heater is employed, the portion of the shield above the asbestos board shall be the same as with the gas burner but the part below the board may be omitted.

Thermometer.

6. *Thermometer.*—The A.S.T.M. Low-Distillation Thermometer shall conform to the following requirements. These specifications cover a total-immersion thermometer graduated in either Centigrade or Fahrenheit degrees, as specified, the ranges being 0 to 300° C. or 30 to 580° F., respectively.

TYPE: Etched stem, glass.
LIQUID: Mercury.
RANGE AND SUBDIVISION: 0 to 300° C. in 1° C. or 30 to 580° F. in 2° F.
TOTAL LENGTH: 378 to 384 mm. (14.88 to 15.12 in.).
STEM: Plain front, enamel back, suitable thermometer tubing.
Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).
BULB: Corning normal or equally suitable thermometric glass.
Length, 10 to 15 mm. (0.39 to 0.59 in.).
Diameter, 5.0 to 6.0 mm. (0.20 to 0.24 in.).
DISTANCE TO 0° C. OR 32° F. LINE FROM BOTTOM OF BULB: 100 to 110 mm. (3.94 to 4.33 in.).
DISTANCE TO 300° C. OR 572° F. LINE FROM TOP OF THERMOMETER: 30 to 45 mm. (1.18 to 1.77 in.).
FILLING ABOVE MERCURY: Nitrogen gas.
TOP FINISH: Glass ring.
GRADUATION: All lines, figures, and letters clear cut and distinct. The first and each succeeding 5° C. or 10° F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.
IMMERSION: Total.
SPECIAL MARKING: "A.S.T.M. Low Distillation," a serial number and the manufacturer's name or trade mark shall be etched on the stem.
SCALE ERROR: The error at any point of the scale when the thermometer is standardized as provided below shall not exceed 0.5° C. or 1° F.
STANDARDIZATION: The thermometer shall be standardized immersed in the testing bath to the top of the mercury column, at the ice point and at temperature intervals of approximately 50° C. or 100° F. up to 300° C. or 572° F.
TEST FOR PERMANENCY OF RANGE: After being subjected to a temperature of 280 to 290° C. or 540 to 560° F. for 24 hours, the accuracy shall be within the limit specified.
CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. Low Distillation, 0 to 300° C." or "A.S.T.M. Low Distillation, 30 to 580° F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the overall length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

7. *Graduate.*—The graduate shall be of the cylindrical type, of uniform diameter, with a pressed or molded base and a lipped top. The cylinder shall be graduated to contain 100 cc., and the graduated portion shall not be less than 7 in. (17.78 cm.) nor more than 8 in. (20.32 cm.) long. It shall be graduated in single cubic centimeters and each fifth mark shall be distinguished by a longer line. It shall be numbered from the bottom up, at intervals of 10 cc. The overall height of the graduate shall not be less than $9\frac{3}{4}$ in. (24.8 cm.), nor more than $10\frac{1}{4}$ in. (26.0 cm.). The graduations shall not be in error by more than 1 cc. at any point on the scale. **Graduate.**

PROCEDURE

8. *Sampling.*—Samples should be collected in a previously cooled bottle, preferably by immersing the bottle in the liquid, where possible, and discarding the first sample. Where immersion is not possible the sample should be drawn off into a previously cooled bottle in such a manner that agitation is kept at a minimum. Immediately close the bottle with a tight-fitting stopper, and place in an ice bath or refrigerator capable of bringing the gasoline to a temperature of not less than 32° F. (0° C.), nor more than 40° F. (4.45° C.). **Sampling.**

9. (*a*) The condenser bath shall be filled with cracked ice[1] and enough water added to cover the condenser tube. The temperature shall be maintained between 32 and 34° F. (0 and 1.1° C.). Agitation with air greatly aids in maintaining the required temperature range. **Procedure.**

(*b*) The condenser tube shall be swabbed to remove any liquid remaining from the previous test. A piece of soft cloth attached to a cord or copper wire may be used for this purpose.

(*c*) One hundred cubic centimeters of the product shall be measured in the 100-cc. graduated cylinder at 32 to 40° F. (0 to 4.45° C.) and transferred directly to the Engler flask. Both the flask and the graduated cylinder shall have been cooled to a temperature of from 32 to 40° F. (0 to 4.45° C.) before use. None of the liquid shall be permitted to flow into the vapor tube.

(*d*) The thermometer provided with a cork shall be fitted tightly into the flask so that it will be in the middle of the neck and so that

[1] Any other convenient cooling medium may be used.

the lower end of the capillary tube is on a level with the inside of the bottom of the vapor outlet tube at its junction with the neck of the flask.

(*e*) The charged flask shall be placed in the $1\frac{1}{4}$-in. (3.175-cm.) opening in the 6 by 6-in. (15.24 by 15.24-cm.) asbestos board with the vapor outlet tube inserted into the condenser tube. A tight connection may be made by means of a cork through which the vapor tube passes. The position of the flask shall be so adjusted that the vapor tube extends into the condenser tube not less than 1 in. (2.54 cm.) nor more than 2 in. (5.08 cm.).

(*f*) The graduated receiver used in measuring the charge shall be placed, without drying, at the outlet of the condenser tube in such a position that the condenser tube shall extend into the graduate at least 1 in. (2.54 cm.), but not below the 100-cc. mark. The graduate shall be immersed at least up to the 100-cc. mark in a transparent bath maintained between the temperatures of 32 and 34° F. (0 and 1.11° C.) The top of the graduate shall be covered closely during the distillation with a piece of blotting paper, or its equivalent, and cut to fit the condenser tube tightly. A lead washer, or other suitable material, resting on the blotting paper is a convenient accessory as it serves to hold the paper tightly against the top of the graduate and also provides the additional weight necessary to overcome the buoyant effect of the liquid in the cooling bath.

10. When everything is in readiness, heat shall be applied at a uniform rate, so regulated that the first drop of the condensate falls from the condenser in not less than two nor more than five minutes. When the first drop falls from the end of the condenser, the receiving cylinder shall be moved so that the end of the condenser tube shall touch the side of the cylinder. The heat shall then be so regulated that the first 10 cc. of distillate shall be recovered in not less than three nor more than four minutes. Thereafter, the rate of distillation shall be maintained uniformly at not less than four nor more than 5 cc. per minute. The reading of the distillation thermometer shall be recorded when the level of the distillate reaches each 10-cc. mark on the graduate.

When the liquid residue in the distillation flask is approximately 5 cc., the heat may be increased because of the presence of heavy ends which have relatively high boiling points. However, no further increase of heat shall be applied after this adjustment. The 4 to 5-cc. rate cannot always be maintained from this point to the end of the distillation, but in no case should the period between the point when approximately 5 cc. of the liquid remains in the flask and the end point be more than 5 minutes.

The heating shall be continued until the mercury reaches a maximum and starts to fall consistently. The highest temperature observed on the distillation thermometer shall be recorded as the end point. Usually this point will be reached after the bottom of the flask has become dry. If the bottom of the flask is not dry, the operator should note this on his report.

The total volume of the distillate collected in the receiving graduate shall be recorded as the "recovery."

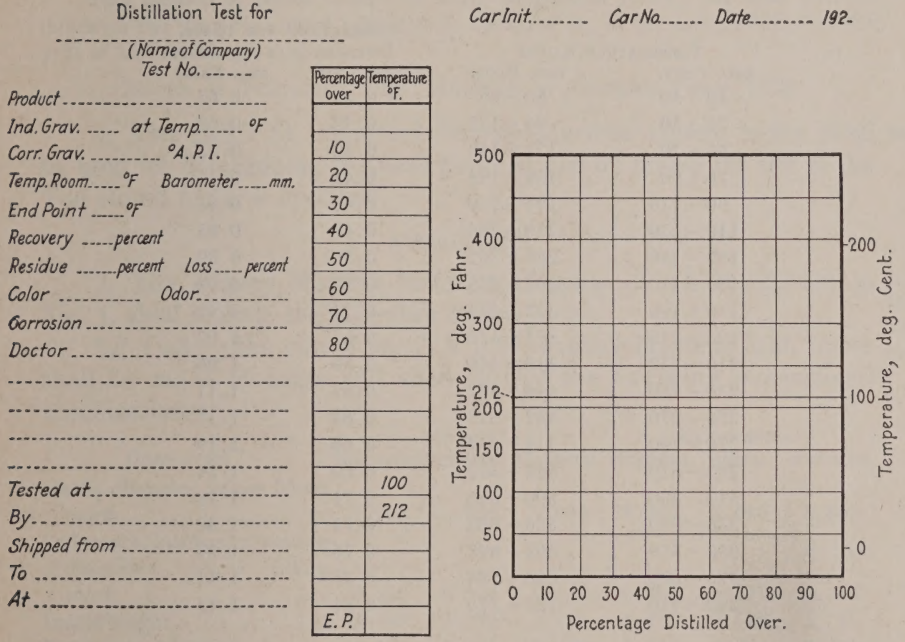


FIG. 3.—Form for Recording Tests on Natural Gas Gasoline.

The cooled residue shall be poured from the flask into a small cylinder graduated in 0.1 cc., measured when cool, and the volume recorded as "residue" at 32 to 40° F. (0 to 4.45° C.).

The difference between 100 cc. and the sum of the recovery and the residue shall be calculated and recorded as "distillation loss."

Correction.

11. *Correction for Barometric Pressure.*—The actual barometric pressure shall be ascertained and recorded, but no correction shall be made except in case of dispute. In such cases the temperature points shall be corrected to 760 mm. (29.92 in.), by the use of the Sydney Young equation, as follows:

For Centigrade readings:

$$C_c = 0.00012\ (760 - P)\ (273 + t_c),$$

For Fahrenheit readings:

$$C_f = 0.00012\ (760 - P)\ (460 + t_f),$$

in which C_c and C_f are, respectively, corrections to be added to the observed temperature t_c or t_f, and P is the actual barometric pressure in millimeters of mercury.

The following table is a convenient approximation of the corrections as calculated by the above equation.

Temperature Range		Correction[1] per 10-mm. Difference in Pressure,	
deg. Cent.	deg. Fahr.	deg. Cent.	deg. Fahr.
10 – 30	50 – 86	0.35	0.63
30 – 50	86 – 122	0.38	0.68
50 – 70	122 – 158	0.40	0.72
70 – 90	158 – 194	0.42	0.76
90 – 110	194 – 230	0.45	0.81
110 – 130	230 – 266	0.47	0.85
130 – 150	266 – 302	0.50	0.89
150 – 170	302 – 338	0.52	0.94
170 – 190	338 – 374	0.54	0.98
190 – 210	374 – 410	0.57	1.02
210 – 230	410 – 446	0.59	1.06
230 – 250	446 – 482	0.62	1.11
250 – 270	482 – 518	0.64	1.15
270 – 290	518 – 554	0.66	1.19
290 – 310	554 – 590	0.69	1.24
310 – 330	590 – 626	0.71	1.28
330 – 350	626 – 662	0.74	1.32
350 – 370	662 – 698	0.76	1.37
370 – 390	698 – 734	0.78	1.41
390 – 410	734 – 770	0.81	1.45

Recording Results.

12. The form shown in Fig. 3 is suggested for the recording of results of tests of natural gas gasoline.

[1] To be added in case barometric pressure is below 760 mm., to be subtracted in case barometric pressure is above 760 mm.

STANDARD METHOD OF TEST
FOR
FLASH POINT BY MEANS OF THE PENSKY-MARTENS CLOSED TESTER[1]

Serial Designation: D 93 – 22

This method is issued under the fixed designation D 93 ; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1922.

1. The A.S.T.M. standard Pensky-Martens closed tester shall be used for determining the flash point of fuel oil unless the use of the Tag closed tester is specified. **Scope.**

APPARATUS

2. The Pensky-Martens tester, a diagram of which appears in Fig. 1, shall include the following major parts: **Pensky-Martens Tester.**

(*a*) *Cup.*—The cup of the A.S.T.M. Pensky-Martens flash tester shall be made of brass and shall satisfy the following dimensional specifications: **Cup.**

Dimensions.	Inches.			Centimeters.		
	Minimum.	Normal.	Maximum.	Minimum.	Normal.	Maximum.
Inside diameter below filling mark	1.950	2.000	2.050	4.953	5.080	5.207
Difference, inside and outside diameters below filling mark	0.120	0.125	0.130	0.305	0.318	0.330
Inside height	2.150	2.200	2.250	5.461	5.588	5.715
Thickness of bottom	0.070	0.095	0.120	0.178	0.241	0.305
Distance from rim to filling mark	0.845	0.860	0.875	2.146	2.184	2.223
Distance lower surface flange to bottom of cup	1.780	1.795	1.810	4.521	4.559	4.597

The inside of the cup may be turned to a slightly larger diameter above the filling mark and the outside may be tapered above the flange but the wall thickness at the upper edge shall be not less than 0.04 in. (0.102 cm.). The flange should be approximately 0.5 in. (1.27 cm.) wide and approximately 0.125 in. (0.318 cm.) thick. It shall be equipped with devices for locating the position of the lid on

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

the cup and the cup in the stove. A handle, attached permanently to the flange of the cup, is a desirable accessory.

Lid. (*b*) *Lid.*—

(*1*) *Stirring Device.*—The lid shall be equipped with a stirring device consisting of a vertical steel shaft, not less than 0.1 in. (0.254

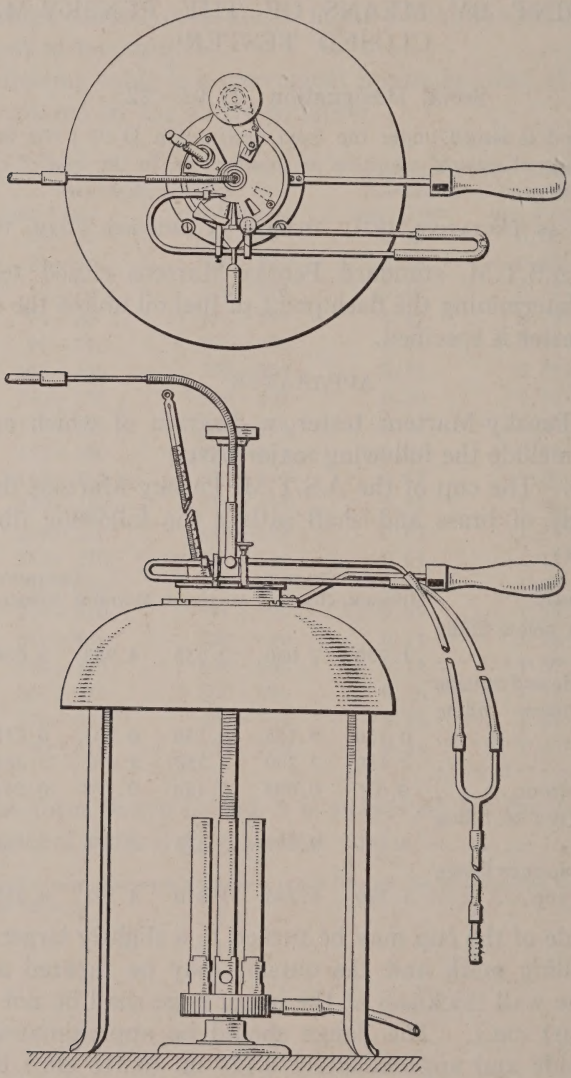
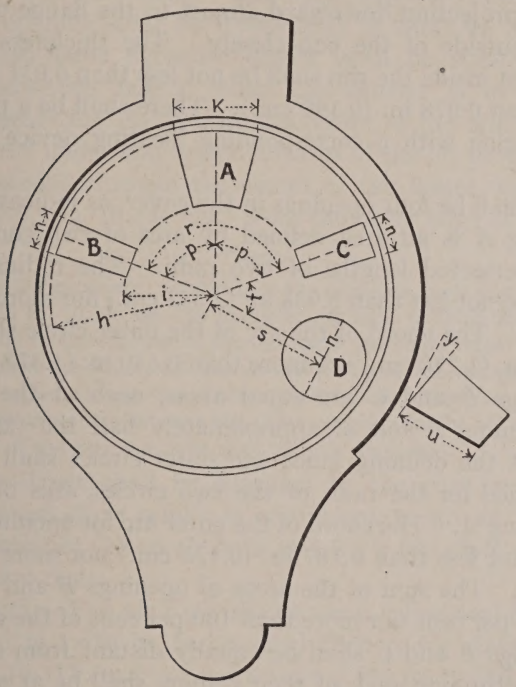


FIG. 1.—The Pensky-Martens Tester.

cm.) nor more than 0.125 in. (0.318 cm.) in diameter, mounted in the center of the cup, and carrying two two-bladed brass propellers.

The blades of both propellers shall be approximately 0.313 in. (0.795 cm.) wide and shall be set at an angle of approximately 45 deg. The smaller (upper) propeller shall have an over-all diameter of approximately 0.75 in. (1.905 cm.). The larger (lower) propeller shall have an over-all diameter between 1.25 and 1.75 in. (3.175 and 4.445 cm.).



h	Minimum 0.938 in,	Maximum 0.969 in.	
i	" 0.531 ",	"	0.563 "
k	" 0.500 ",	"	0.540 "
n	" 0.187 ",	"	0 219 "
s	Approximately 0.75 in.		
u	" 0.5 ".		
Angles *p* -	Equal		
Angle *r* -	Min 135°;	Max.	140°
" *t* -	" 50°;	"	60°
" *y* -	" 10°;	"	15°

FIG. 2.—Cover for Pensky-Martens Tester.

The thickness of the propeller blades shall be not less than 0.057 in. (0.145 cm.) nor more than 0.081 in. (0.206 cm.), which limits correspond respectively to No. 15 and No. 12 B. and S. gage sheet brass. The collars on which the propeller blades are mounted shall have horizontal and vertical dimensions not greater than 0.4 in. (1.016 cm.).

The plane of the center of the upper propeller shall be 0.4 in. (1.016 cm.) below the level of the rim of the cup. The plane of the

center of the lower propeller shall be 2.0 in. (5.08 cm.) below the level of the rim of the cup. The level of the rim of the cup is in effect the level of the plane part of the portion of the lower surface of the lid inside the rim.

(*2*) *Cover proper.*—The cover proper shall be of brass and shall have a rim projecting downward almost to the flange of the cup and fitting the outside of the cup closely. The thickness of the cover, measured just inside the rim shall be not less than 0.031 in. (0.079 cm.) nor more than 0.078 in. (0.198 cm.). There shall be a proper locating device engaging with a corresponding locating device on the flange of the cup.

There shall be four openings in the cover, as indicated in Fig. 2.

Opening *A* is an area defined by arcs of two concentric circles and the intersected lengths of two radii. The radius of the outer circle shall be not less than 0.938 in. (2.383 cm.) nor more than 0.969 in. (2.461 cm.). The chord of the arc of the outer circle shall be not less than 0.500 in. (1.270 cm.) nor more than 0.540 in. (1.372 cm.).

Openings *B* and *C* are equal areas, each of the same general form as opening *A* but of approximately half the (angular) width. The radii of the defining inner and outer circles shall be within the limits specified for the radii of the two circles, arcs of which partly define opening *A*. The chord of the outer arc for opening *B* or opening *C* shall be not less than 0.187 in. (0.475 cm.) nor more than 0.219 in. (0.556 cm.). The sum of the areas of openings *B* and *C* shall be not less than 75 per cent nor more than 100 per cent of the area of opening *A*. Openings *B* and *C* shall be equally distant from opening *A* and radii drawn through each of their centers shall be at an angle of not less than 135 deg. nor more than 140 deg.

Openings *A*, *B*, and *C* need not conform exactly to the shape of geometrical figures bounded by arcs of two concentric circles and intersected lengths of radii. Their boundaries must, however, fall on or between the lines indicated by the limiting values of the dimensional specification of the preceding text and of Fig. 2.

Opening *D* is for a thermometer collar. Its center is approximately 0.75 in. (1.905 cm.) from the center of the lid and on a radius at an angle of not less than 50 deg. nor more than 60 deg. from a radius passing through the center of opening *C*. The thermometer collar shall have an inside diameter of approximately 0.5 in. (1.27 cm.). It shall be set at an angle of not less than 10 deg. nor more than 15 deg. from the perpendicular.

(*3*) *Shutter.*—The lid shall be equipped with a brass shutter, approximately 0.094 in. (0.239 cm.) thick operating on the plane of

the upper surface of the lid. The shutter shall be so shaped and mounted that it rotates on the axis of the horizontal center of the lid between two stops so placed that when in one extreme position the openings *A*, *B*, and *C* of the lid are completely closed and when in the other extreme position these orifices are completely opened.

(*4*) *Flame exposure device.*—The flame exposure device shall have a tip with an opening 0.027 in. (0.069 cm.) to 0.031 in. (0.079 cm.) in diameter. The flame exposure device shall be equipped with an operating mechanism which, when the shutter is in the "open" position, depresses the tip so that the center of the orifice is between the planes of the under and upper surfaces of the lid proper at a point on a radius passing through the center of the larger opening *A* and approximately 0.1 in. (0.254 cm.) from the outer edge of the opening.

NOTE.

A pilot flame for automatic relighting of the exposure flame should be provided.

A bead 0.156 in. (0.396 cm.) in diameter, of some suitable material, may be mounted on the lid so that the size of the test flame can be regulated by comparison.

The mechanism operating the shutter should be of the spring type and constructed so that when at rest the shutter shall exactly close the three openings. When operated to the other extreme the three openings in the lid shall be exactly open and the tip of the exposure tube shall be fully depressed.

(*c*) *Stove.*—Heat shall be supplied to the cup by means of a properly designed stove which is equivalent to an air bath. This stove shall consist of (1) an air bath and (2) a top plate on which the flange of the cup rests. Stove.

(*1*) *Air bath.*—The air bath shall have a cylindrical interior 1.625 in. (4.128 cm.) to 1.656 in. (4.206 cm.) deep and a diameter not less than 0.125 in. (0.317 cm.) nor more than 0.156 in. (0.396 cm.) greater than the outside diameter of the cup. The air bath may be either a flame heated metal casting or an electric resistance element.

NOTE.

If the heating element is a flame heated metal casting it shall be so designed and used that the temperature of bottom and walls is approximately the same. On this account it should be not less than 0.25 in. (0.635 cm.) thick. The casting shall be designed so that products of combustion of the flame cannot pass up and in contact with the cup.

If the air bath is of the electric resistance type it shall be constructed so that all parts of the interior surface are heated equally. This necessitates an even distribution of resistance wire over bottom and walls and a method of construction such that heat is given out from the whole core of the resistance element rather than directly from the wire.

(*2*) *Top plate.*—The top plate shall be of metal. The total distance from the upper surface of the plate to the bottom of the air bath shall exceed the distance from the under surface of the flange to

the bottom of the cup by not less than 0.063 in. (0.160 cm.) nor more than 0.125 in. (0.317 cm.).

The top plate shall be mounted with an air gap between it and the air bath. The top plate may be attached to the air bath by means of three screws and spacing bushings. The spacing bushings should be of proper thickness to define the air gap which shall be not less than 0.125 in. (0.317 cm.) nor more than 0.187 in. (0.475 cm.). The spacing bushings shall be not more than 0.375 in. (0.952 cm.) in diameter.

Thermometers.

(*d*) *Thermometers.*—Two standard thermometers shall be used with the A.S.T.M. Pensky-Martens tester. The low range, "P. M. and Tag" thermometer shall be used for tests when the indicated reading falls within the limits 20 to 200° F. The "P.M. high" thermometer shall be used for tests when the indicated reading falls within the limits 230 to 700° F. For the range 200 to 230° F. either thermometer may be employed, depending on the convenience of the operator. The thermometers shall comply with the requirements given in Tables I and II. The Low Range "P. M. and Tag" thermometer specifications cover a special thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges −7 to +110° C. or +20 to +230° F., respectively. The High Range "P. M. High" thermometer specifications cover a special thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being 90 to 370° C. or 200 to 700° F., respectively.

Thermometers shall be mounted so that the bottom of the bulb is 1.75 in. (4.445 cm.) below the level of the rim of the cup (which corresponds to the level of the lower surface of the portion of the lid inside the rim).

TABLE I.—SPECIFICATIONS FOR LOW RANGE "P. M. AND TAG" THERMOMETER.[1]

TYPE: Etched stem, glass.

LIQUID: Mercury.

RANGE AND SUBDIVISION: −7 to +110° C. in 0.5° C. or +20 to +230° F. in 1° F.

TOTAL LENGTH: 273 to 277 mm. (10.75 to 10.92 in.).

STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).

BULB: Corning normal or equally suitable thermometric glass.
Length, 9 to 13 mm. (0.35 to 0.51 in.).
Diameter, not greater than stem.

DISTANCE TO −7° C. OR +20° F. LINE FROM BOTTOM OF BULB: 75 to 90 mm. (2.93 to 3.54 in.).

[1] The low range "P.M. and Tag" thermometer is the same instrument as that specified for use with the Tag closed tester, see the Standard Method of Test for Flash Point of Volatile Flammable Liquids (Serial Designation: D 56) of the American Society for Testing Materials, p. 300.

DISTANCE TO 110° C. OR 230° F. LINE FROM TOP OF THERMOMETER: 25 to 40 mm. (0.98 to 1.57 in.).

EXPANSION CHAMBER: To permit heating the thermometer at least 50° C. (90° F.) above highest temperature on scale.

FILLING ABOVE MERCURY: Nitrogen gas.

TOP FINISH: Glass ring.

GRADUATION: All lines, figures, and letters clear cut and distinct. The whole degree Centigrade lines or the first and each succeeding 5° F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 5° C. or 10° F.

IMMERSION: 57 mm. or 2¼ in. The words "57-mm. immersion" on Centigrade or "2¼-in. immersion" on Fahrenheit thermometers and a line around the stem 57.0 mm. or 2.25 in. above the bottom of the bulb shall be etched on the thermometer.

SPECIAL MARKING: "A.S.T.M. P.M. and Tag.," a serial number and the manufacturer's name or trade mark, shall be etched on the stem.

SCALE ERROR: The error at any point of the scale when the thermometer is standardized as provided below shall not exceed 0.5° C. or 1° F., respectively.

STANDARDIZATION: The thermometer shall be standardized at the ice point and at intervals of approximately 30° C. or 50° F. for 57-mm. or 2¼-in. immersion and for the following temperatures of the emergent mercury column:

THERMOMETER READING	AVERAGE TEMPERATURE OF EMERGENT MERCURY COLUMN
20° C. 70° F.	20° C. 70° F.
40° C. 100° F.	31° C. 86° F.
70° C. 150° F.	40° C 104° F.
100° C. 212° F.	48° C. 118° F.

CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. P.M. and Tag., −7 to +110° C." or "A.S.T.M. P.M. and Tag., +20 to +230° F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

TABLE II.—SPECIFICATIONS FOR HIGH RANGE "P. M. HIGH" THERMOMETER.

TYPE: Etched stem, glass.

LIQUID: Mercury.

RANGE AND SUBDIVISION: 90 to 370° C. in 2° C. or 200 to 700° F. in 5° F.

TOTAL LENGTH: 273 to 277 mm. (10.75 to 10.92 in.).

STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).

BULB: Corning normal or equally suitable thermometric glass.

Length, not over 10 mm. (0.39 in.).

Diameter, not greater than stem.

DISTANCE TO 93° C. OR 200° F. LINE FROM BOTTOM OF BULB: 75 to 90 mm. (2.93 to 3.54 in.).

DISTANCE TO 371° C. OR 700° F. LINE FROM TOP OF THERMOMETER: 25 to 40 mm. (0.98 to 1.57 in.).

FILLING ABOVE MERCURY: Nitrogen gas.

TOP FINISH: Glass ring.

GRADUATION: All lines, figures, and letters clear cut and distinct. The first and each succeeding 10° C. or 25° F. line to be longer than the remaining lines. Graduations to be numbered at multiples of 20° C. or 50° F.

IMMERSION: 57 mm. or 2¼ in. The words "57-mm. immersion" on Centigrade thermometers or "2¼-in. immersion" on Fahrenheit thermometers and a line around the stem 57.0 mm. or 2.25 in. above the bottom of the bulb shall be etched on the thermometer.

SPECIAL MARKING: "A.S.T.M. P.M. High," a serial number and the manufacturer's name or trade mark shall be etched on the stem.

SCALE ERROR: The error at any point of the scale when the thermometer is standardized as described below shall not exceed 1° C. or 2½° F.

STANDARDIZATION: The thermometer shall be standardized at intervals of approximately 50° C. or 100° F. for 57-mm. or 2¼-in. immersion and for the following temperatures of the emergent mercury column:

THERMOMETER READING	AVERAGE TEMPERATURE OF EMERGENT MERCURY COLUMN
100° C. 200° F.	61° C. 140° F.
150° C. 300° F.	65° C. 149° F.
200° C. 400° F.	71° C. 160° F.
250° C. 500° F.	78° C. 175° F.
300° C. 600° F.	87° C. 195° F.
350° C. 700° F.	99° C. 220° F.

TEST FOR PERMANENCY OF RANGE: After being subjected to a temperature between 360 and 370° C. or 680 and 700° F. for 24 hours, the accuracy shall be within the limit specified.

CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. P.M. High, 90 to 370° C." or "A.S.T.M. P.M. High, 200 to 700° F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

PROCEDURE

Procedure.

3. (*a*) All parts of the cup and its accessories shall be thoroughly clean and dry before the test is started. Particular care shall be taken to avoid the presence of any gasoline or naphtha used to clean the apparatus after a previous test.

(*b*) The cup shall be filled with the oil to be tested up to the level indicated by the filling mark.

(*c*) The lid shall be placed on the cup and the latter set in the

stove. Care shall be taken to have the locating devices properly engaged. The thermometer shall be inserted. If it is known that the oil will flash above 220° F. the "P.M. high" thermometer may be selected; otherwise, it is preferable to start with the "P.M. and Tag" thermometer and change in case a temperature of 220 to 230° F. is reached.

(*d*) The test flame shall be lighted and adjusted so that it is of the size of a bead $\frac{5}{32}$ in. (3.97 mm.) in diameter.

(*e*) Heat shall be supplied at such a rate that the temperature read on the thermometer increases not less than 9 nor more than 11° F. per minute. The stirrer shall be turned at a rate of from 1 to 2 revolutions per second.

(*f*) Application of the test flame shall be made at each temperature reading which is a multiple of 2° F. up to 220° F. For the temperature range above 220° F., application shall be made at each temperature reading which is a multiple of 5° F. The first application of the test flame shall be made at a temperature at least 30° F. below the actual flash point. Application of the test flame shall be made by operating the device controlling the shutter and test flame burner so that the flame is lowered in one-half second, left in its lowered position for one second, and quickly raised to its high position. Stirring shall be discontinued during the application of the test flame.

Flash Point.

4. The flash point is taken as the temperature read on the thermometer at the time of the flame application that causes a distinct flash in the interior of the cup. The true flash must not be confused with the bluish halo that sometimes surrounds the test flame for the applications preceding the one that causes the actual flash.

Barometric Pressure.

5. The barometric pressure shall be observed and recorded. No corrections shall be made except in case of dispute when the flash point figure shall be corrected according to the following rule:

For each inch (25 mm.) below 29.92 in. (760 mm.) barometric reading add 1.6° F. to the flash point.

For each inch (25 mm.) above 29.92 in. (760 mm.) barometric reading subtract 1.6° F. from the flash point.

STANDARD METHOD OF TEST
FOR
FLASH AND FIRE POINTS BY MEANS OF OPEN CUP[1]

Serial Designation: D 92 – 24

This method is issued under the fixed designation D 92; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921: ADOPTED IN AMENDED FORM, 1924.

Scope.

1. The open cup flash and fire test on all products except fuel oils and those having an open cup flash below 175° F. shall be determined in the Cleveland Open Cup.

APPARATUS

Flash Cup.

2. The cup shall be supported by a metal plate $\frac{1}{4}$ in. (0.635 cm.) in thickness and 6 in. (15.24 cm.) in width. The plate shall be of brass, cast iron, wrought iron or steel. In the center of the plate there shall be a plane depression $\frac{1}{32}$ in. (0.079 cm.) in depth, and of just sufficient diameter to fit the cup. There shall be a circular opening $2\frac{3}{16}$ in. (5.50 cm.) in diameter, cut through the plate, centering with the center of the above-mentioned depression. The plate shall be covered with a sheet of hard asbestos board $\frac{1}{4}$ in. in thickness, and of the same shape as the metal plate. There shall be cut in the center of the asbestos board a circular hole just fitting the cup. Heat may be supplied from any convenient source. The use of a gas burner, electric heater, or alcohol lamp is permitted, but under no circumstances are products of combustion or free flame allowed to come up around the cup. The source of heat shall be centered under the opening in the plate and shall be of a type that will not produce local superheating. If a flame heater is used, it may be protected from drafts or excessive radiation by any suitable type of shield, that does not project above the level of the upper surface of the asbestos board.

Thermometer.

3. The thermometer shall conform to the following requirements. These specifications cover a special thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being −6 to +400° C., or +20 to +760° F., respectively.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

TYPE: Etched stem, glass.
LIQUID: Mercury.
RANGE AND SUBDIVISION: −6 to +400° C. in 2° C. or +20 to +760° F. in 5° F.
TOTAL LENGTH: 303 to 307 mm. (11.92 to 12.08 in.).
STEM: Plain front, enamel back, suitable thermometer tubing.
Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).
BULB: Corning normal or equally suitable thermometric glass.
Length, not over 13 mm. (0.51 in.).
Diameter, not greater than stem.

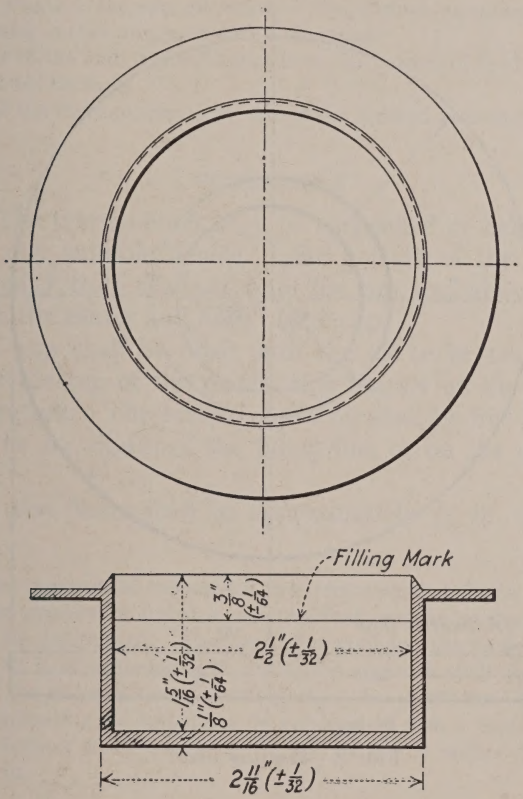


FIG. 1.—Cleveland Open Cup

DISTANCE TO −6° C. OR 20° F. LINE FROM BOTTOM OF BULB: 40 to 50 mm. (1.57 to 1.97 in.).
DISTANCE TO 400° C. OR 760° F. LINE FROM TOP OF THERMOMETER: 30 to 45 mm. (1.18 to 1.77 in.).
FILLING ABOVE MERCURY: Nitrogen gas.
TOP FINISH: Red glass ring.
GRADUATION: All lines, figures, and letters clear cut and distinct. The first and each succeeding 10° F. line to be longer than the intermediate lines. Graduations to be numbered at each multiple of 20°.

IMMERSION: 25 mm. or one inch. The words "1-in. immersion" on Fahrenheit thermometers or "25 mm. immersion" on Centigrade thermometers and a line around the stem 25.4 mm. or 1.00 in. above the bottom of the bulb shall be etched on the thermometer.

SPECIAL MARKING: "A.S.T.M. Open Flash" a serial number and the manufacturer's name or trade mark shall be etched on the stem.

SCALE ERROR: The error at any point of the scale when the thermometer is standardized as provided below shall not exceed 1° C. up to 372° C. or 2½° F. up to 700° F.

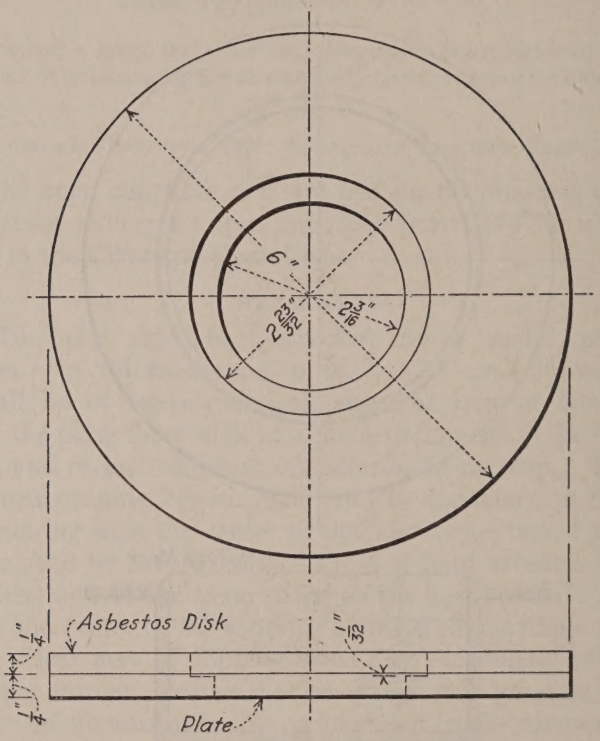


FIG. 2.—Heating Plate

STANDARDIZATION: The thermometer shall be standardized at the ice point and at intervals of approximately 100° F. for 25-mm. or 1-in. immersion and for the following temperatures of the emergent mercury column:

THERMOMETER READING	AVERAGE TEMPERATURE OF EMERGENT MERCURY COLUMN
100° C. 200° F.	44° C. 110° F.
150° C. 300° F.	54° C. 129° F.
200° C. 400° F.	64° C. 150° F.
250° C. 500° F.	77° C. 175° F.
300° C. 600° F.	91° C. 205° F.
350° C. 700° F.	108° C. 240° F.

TEST FOR PERMANENCY OF RANGE: After being subjected to a temperature between 360 to 370° C. or 680 to 700° F. for 24 hours, the accuracy shall be within the limit specified.

CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. Open Flash, −6 to +400° C." or "A.S.T.M. Open Flash, +20 to +760° F.," according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

PROCEDURE

Procedure.

4. (*a*) The thermometer shall be suspended or held in a vertical position by any suitable device. The bottom of the bulb shall be $\frac{1}{4}$ in. (0.635 cm.)[1] from the bottom of the cup, and above a point half way between the center and back of the cup.

(*b*) The cup shall be filled with the oil to be tested in such a manner that the top of the meniscus is exactly at the filling line at room temperature. The surface of the oil shall be free from bubbles. There shall be no oil above the filling line or on the outside of the apparatus.

(*c*) The test flame shall be approximately $\frac{5}{32}$ in. (0.397 cm.) in diameter.

NOTE.—For purposes of comparison it is recommended that a bead of suitable light colored material be mounted in a convenient position so that the size of the test flame can be determined. The device for applying the flame may be of any suitable type but it is suggested that the tip be approximately $\frac{1}{16}$ in. (0.159 cm.) in diameter at the end and that the orifice be $\frac{1}{32}$ in. (0.079 cm.) in diameter. If the device for operating the test flame be mounted in such a manner as to permit automatic duplication of the sweep of the test flame the radius of swing shall be not less than 6 in.

(*d*) The test flame shall be applied as the temperature read on the thermometer reaches each successive 5° F. mark. The flame shall pass in a straight line (or on the circumference of a circle having a radius of at least 6 in.) across the center of the cup and at right angles to the diameter passing through the thermometer. The test flame shall, while passing across the surface of the oil, be in the plane of the upper edge of the cup. The time for the passage of the test flame across the cup shall be approximately one second.

[1] The immersion line engraved on the thermometer stem will be $\frac{1}{16}$ in. (0.159 cm.) below the level of the rim of the cup.

(*e*) The oil shall be heated at a rate not exceeding 30° F. per minute temperature rise, till a point is reached approximately 100° F. below the probable flash point of the oil. Thereafter the rate of heating shall be decreased and for at least the last 50° F. before the flash point is reached, the rate shall be not less than 9 nor more than 11° F. per minute.

Flash Point. 5. The flash point shall be taken as the temperature read on the thermometer when a flash appears at any point on the surface of the oil. The true flash must not be confused with a bluish halo that sometimes surrounds the test flame.

Fire Point. 6. After determining the flash point, the heating shall be continued at the specified rate of 9 to 11° F. per minute, and application of the test flame shall be made at the specified intervals until the oil ignites and continues to burn for a period of at least 5 seconds. The method of application of the flame shall be the same or for flash point. The temperature read at the time of the flame application which causes burning for a period of 5 seconds or more shall be recorded as the fire point.

Precautions. 7. The flash point and fire point tests shall be made in a room or compartment free from air drafts. The operator shall avoid breathing over the surface of the oil. It is desirable that the room or compartment be darkened sufficiently so that the flash may be readily discernible.

STANDARD METHOD OF TEST
FOR
MELTING POINT OF PARAFFIN WAX[1]

Serial Designation: D 87 – 22

This method is issued under the fixed designation D 87; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED, 1922.

DEFINITION

Definition.

1. *A.S.T.M. Paraffin Wax Melting Point.*—The A.S.T.M. Paraffin Wax Melting Point is the temperature at which melted paraffin wax, when allowed to cool under definite prescribed conditions, first shows a minimum rate of temperature change.

NOTE.—The so-called "American Melting Point" is an arbitrary figure 3° F. higher than the A.S.T.M. Paraffin Wax Melting Point.

APPARATUS

Containers.

2. *Wax Container.*—Test tube of standard form, 25 mm. (1 in.) outside diameter and 100 mm. (4 in.) long. It may be marked with a filling line, 2 in. above the bottom. This test tube shall be closed in a tightly fitting cork having two openings, one at the center for the melting point thermometer and the other for a stirrer at one side of the center. The opening for the stirrer may be lined with glass or metal tubing to act as a guide for the stirrer.

Air Bath.

3. *Air Bath.*—Suitable water-tight cylinder, 2 in. in inside diameter and $4\frac{1}{2}$ in. deep. This air bath shall be provided with a tightly fitting cork having a central opening for holding the test tube firmly in a vertical position in the center of the air bath.

Water Bath.

4. *Water Bath.*—Suitable cylinder, $5\frac{1}{8}$ in. in inside diameter and 6 in. deep. This water bath shall be provided with a suitable cover and with the guides and fasteners necessary to hold the air bath firmly in a vertical central position so that the sides and bottom of the air bath shall be surrounded by a layer of water $1\frac{1}{2}$ in. thick. The water bath cover shall have a slot for introduction of a suitable stirrer and shall have an opening for the bath thermometer so that the latter

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

may be suspended in a vertical position $\frac{3}{4}$ in. from the outside wall of the water bath. Air bath, water bath and water bath cover may be conveniently made of metal in one assembly as shown in Fig. 1.

Stirrer.

5. *Stirrer in test tube.*—Brass or copper wire, $\frac{1}{8}$ in. in diameter and about 12 in. long. A circular loop, $\frac{1}{2}$ in. in diameter, shall be formed at one end of this wire in such a manner that the loop lies in a hor-

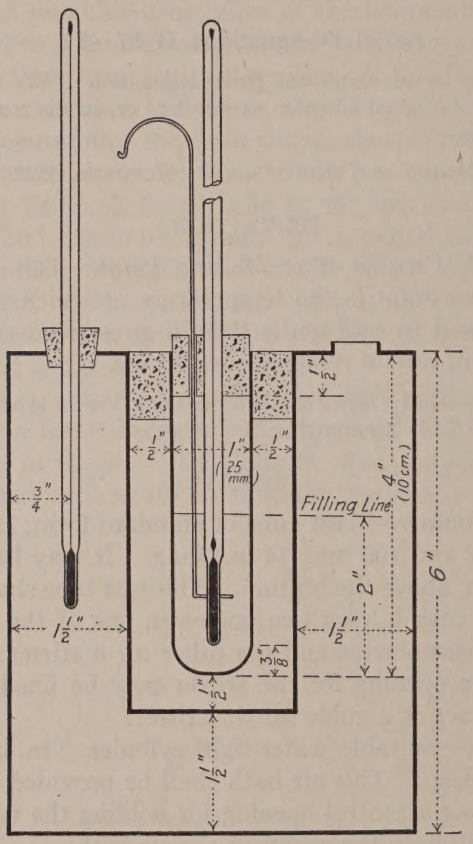


FIG. 1.—Apparatus for Determination of Melting Point of Paraffin Wax.

izontal plane when the rest of the wire is in a vertical position. The stirrer thus formed shall be passed through the proper opening in the test-tube cork and the upper end may then be bent into a shape convenient for holding.

Thermometer.

6. *Thermometer.*—The A.S.T.M. Paraffin Wax Melting Point thermometer shall conform to the following requirements. These speci-

fications cover a special thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being 27 to 71° C. or 80 to 160° F., respectively.

TYPE: Etched stem, glass.
LIQUID: Mercury.
RANGE AND SUBDIVISION: 27 to 71° C. in 0.1° C. or 80 to 160° F. in 0.2° F.
TOTAL LENGTH: 365 to 371 mm. (14.38 to 14.56 in.).
STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).
BULB: Corning normal or equally suitable thermometric glass.
Length, not over 28 mm. (1.10 in.).
Diameter, not greater than stem.
DISTANCE TO 27° C. OR 80° F. LINE FROM BOTTOM OF BULB: 105 to 115 mm. (4.13 to 4.53 in.).
DISTANCE TO 71° C. OR 160° F. LINE FROM TOP OF THERMOMETER: 25 to 40 mm. (0.98 to 1.57 in.).
CONTRACTION CHAMBER: Top to be not more than 41 mm. (1.61 in.) above bottom of bulb.
EXPANSION CHAMBER: To permit heating the thermometer to at least 100° C. (212° F.).
FILLING ABOVE MERCURY: Nitrogen gas.
TOP FINISH: Plain.
GRADUATION: All lines, figures, and letters clear cut and distinct. The first and each succeeding fifth line to be longer than the remaining lines. Graduations to be numbered at each 1° C. or each multiple of 2° F.
IMMERSION: 79 mm. or $3\frac{1}{8}$ in. The words "79-mm. immersion" on Centigrade thermometers or "$3\frac{1}{8}$-in. immersion" on Fahrenheit thermometers, and a line around the stem 79.0 mm. or 3.13 in. above the bottom of the bulb shall be etched on the stem.
SPECIAL MARKING: "A.S.T.M. Pffe. M.P.," a serial number and the manufacturer's name or trade mark shall be etched on the stem.
SCALE ERROR: The error at any point of the scale when the thermometer is standardized as provided below shall not exceed 0.1° C. or 0.2° F., respectively.
STANDARDIZATION: The thermometer shall be standardized at intervals of approximately 10° C. or 20° F. for 79-mm. or $3\frac{1}{8}$-in. immersion and for an average temperature of the emergent mercury column of 25° C. or 77° F.
CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. Pffe. M.P., 27 to 71° C." or "A.S.T.M. Pffe. M.P., 80 to 160° F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

Bath Thermometer.

7. Bath thermometer of any suitable type, accurate to 2° F. throughout the required range.

PROCEDURE

Procedure.

8. An average sample of the wax to be tested shall be melted in a suitable container in a water bath whose temperature shall be not more than 35° F. above the approximate melting point of the wax sample. Direct heat, such as a flame or hot plate, shall not be used and the wax sample shall not be held in the melted condition any longer than necessary.

The test tube shall be filled with melted wax to a height of 2 in. The test-tube cork, carrying the stirrer and the melting point thermometer with the $3\frac{1}{8}$-in. immersion line at the under surface of the cork, shall be inserted into the test tube for a distance of $\frac{1}{2}$ in. The lower end of the thermometer bulb shall then be $\frac{3}{8}$ in. from the bottom of the test tube.

The air bath being in its proper position in the water bath, the latter shall be filled to within $\frac{1}{2}$ in. of the top with water at a temperature 15 to 20° F. below the approximate melting point of the wax sample.

The test tube containing the melted wax, with wax stirrer and thermometer in place, shall be inserted into the air bath in a central vertical position so that the bottom of the test tube is $\frac{1}{2}$ in. from the bottom of the air bath. The temperature of the water bath shall be adjusted by stirring if necessary, so that it shall be lower than the temperature of the wax sample by not more than 30° F. and not less than 25° F., when the wax sample has cooled to a temperature 10° F. above its approximate melting point.

When these conditions have been obtained, temperature adjustment and stirring of the water bath shall be discontinued. The wax shall be stirred continuously during the remainder of the test, the stirring loop being moved up and down throughout the entire length of the test tube in a steady motion at the rate of 20 complete cycles per minute. The melting point thermometer reading, estimated to 0.1° F. shall be observed and recorded every 30 seconds. The temperature of the wax will fall gradually at first, will then become almost constant and will then again fall gradually.

The melting point thermometer reading, estimated to 0.1° F., shall be observed and recorded every 30 seconds, for at least three minutes after the temperature again begins to fall after remaining almost constant. The record of temperature readings shall then be inspected and the average of the first four readings that lie within a range of 0.2° F. shall be considered as the uncorrected melting point. This temperature shall be corrected if necessary for error in the

thermometer scale and the corrected temperature shall be reported as the "A.S.T.M. Paraffin Wax Melting Point."

ACCURACY

9. Duplicate determinations on the same sample should differ by not more than 0.2° F. Accuracy.

STANDARD METHOD OF TEST
FOR
STEAM EMULSION OF LUBRICATING OILS[1]

Serial Designation: D 157 - 27

This method is issued under the fixed designation D 157; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED IN AMENDED FORM, 1927.

Scope.

1. This method shall be used on all oils when an emulsion, demulsibility or emulsification test is required. This method is commonly used for turbine oils and may be used for other lubricating oils.

Description.

2. The Steam Emulsion Number (A.S.T.M. S.E.) is the number of seconds required for an oil to separate when emulsified and separated under definitely prescribed conditions.

APPARATUS

Steam Generator.

3. The steam generator shall be made of either metal or glass of at least one-liter capacity, capable of withstanding the heat necessary for continued use in the production of steam. It shall be fitted with three outlets with suitable connections for rubber tubing. In case of a metal generator, a large opening for filling and a suitable water gage shall be a necessary part of the apparatus.

Baths.

4. The baths shall be either of metal or glass, with a capacity of 3 to $3\frac{1}{2}$ liters and a depth of $7\frac{1}{2}$ to 9 in. A good quality battery jar or beaker is entirely satisfactory. A glass bath shall be used for emulsification and a metal or glass bath may be used for the separation.

Source of Heat.

5. Heat for the steam generator shall be supplied by a suitable gas burner or electric hot plate. The separating bath may be heated by any convenient means, including an auxiliary steam line as shown in Fig. 1.

Oil Container.

6. The oil container shall be a 25 by 200-mm. test tube graduated from zero or from 10 to 50 cc. in cubic centimeters, each even 5-cc. line to encircle the tube.

Steam Piping.

7. The steam piping or the steam delivery tube shall consist of a piece of thin-wall glass tubing, not less than 2.3 nor more than 2.7 mm. in inside diameter and 12 in. in length. The steam pipe shall be

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

cut off diagonally at an angle of 30 deg. with the axis of the tube at the discharge orifice and shall be bent at right angles 10 in. from the discharge orifice.

8. Accessories shall consist of: **Accessories.**

(*a*) Suitable wooden or metal frames or holders for holding all containers in a vertical position in the baths.

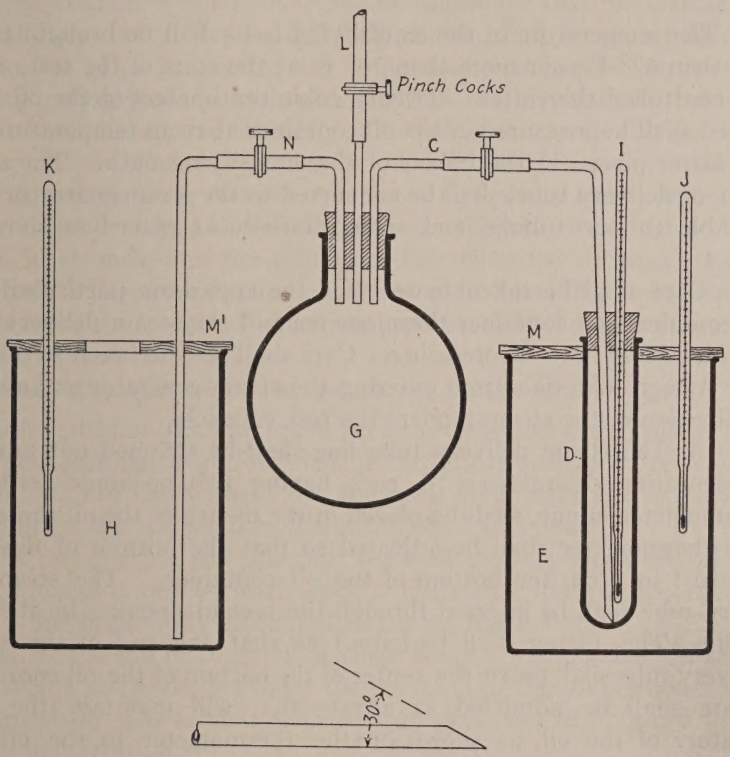


FIG. 1.—Apparatus for Steam Emulsion Test, Self-Generating Steam Supply.

(*b*) Thermometers for the separating and emulsifying baths (floating type thermometers of suitable range);

(*c*) Thermometers for the oil container tube (engraved-stem type, of suitable range, graduated in 1° F., 5 to 7 mm. in diameter);

(*d*) Corks, rubber tubing and screw pinch cocks.

PROCEDURE

9. The apparatus shall be assembled as shown in Fig. 1. The steam generator shall be filled one-half full of water and heat applied. **Preparation.**

The baths shall be filled with 3 liters ±60 cc. of water. The temperature in the separating bath shall be raised to and maintained at 200 to 203° F.

NOTE.—Care must be taken if glass battery jars are used, as direct heating by flame or electric hot point may cause breakage. Use of steam in this connection insures against breakage.

The temperature of the emulsifying bath shall be brought to not less than 67° F., nor more than 78° F. at the start of the test, and is not controlled thereafter. Twenty cubic centimeters of the oil to be tested shall be measured in the oil container at room temperature and the latter placed in the holder of the emulsifying bath. The steam pipe, or delivery tube, shall be connected to the steam generator with suitable rubber tubing, and screw pinch-cocks placed as shown in Fig. 1.

Care shall be taken to see that the apparatus, particularly the oil container, oil container thermometer and the steam delivery tube are *chemically clean* before using. Care shall also be taken to prevent any foreign materials from entering the steam generator as any contamination of the steam renders the test valueless.

Emulsification.

10. The steam delivery tube line shall be steamed out until all condensation disappears. A cork having two openings, with the thermometer in one, shall be placed in the mouth of the oil container. The thermometer shall be adjusted so that the bottom of the bulb is $\frac{3}{4}$ to 1 in. from the bottom of the oil container. The steam delivery tube shall be inserted through the second opening in the cork (Note.—This fitting shall be loose.) so that the end of the steam delivery tube shall touch the center of the bottom of the oil container. Steam shall be admitted at a rate that will maintain the temperature of the oil, as shown on the thermometer in the oil container, between 190 and 195° F.

NOTE.—The usual time necessary for the temperature of the oil to come to this point is 45 to 75 seconds, depending on its character.

This control shall be effected by manipulation of the pinch-cocks on the steam delivery line and steam exhaust line from the steam generator. The steam supply shall be sufficient at all times to cause a generous discharge from the exhaust line. Steaming shall be continued until the volume of condensed steam and oil in the oil container tube is 40 cc. ±3 cc. The time required for this operation shall be 4 to 6.5 minutes, depending on the quality of the oil, altitude, etc. If condensed water amounts to 20 cc. in less than 4 minutes, it shall

be taken as an indication of wet steam or incomplete steaming out of the line and the test shall be re-run.

NOTE.—The apparent volume in the tube near the end of the steaming operation is approximately 12 to 15 cc. greater than the actual volume due to displacement caused by steam, thermometer and steam delivery tube.

Separation.

11. The steam delivery tube shall be withdrawn as soon as the required volume is obtained. The oil container shall be transferred immediately to the separating bath which shall be maintained at 200 to 203° F. It is extremely important that the temperature of the separating bath be maintained within the given limits. A stop watch shall be started at the instant of withdrawal of the steam delivery tube. The cork containing the thermometer shall be removed after the oil container and contents have been placed in the separating bath. The contents of the oil container shall be examined at least every 30 seconds, and the volume of the separated oil layer shall be recorded. This examination shall be made by withdrawing the oil container and its contents from the separating bath, and reading the volumes.

NOTE.—This operation shall not require over five seconds.

No differentiation shall be made between clear and turbid oil.

This reading operation shall be continued until 20 cc. of oil have separated. In cases where the 20-cc. oil layer does not separate in 20 minutes, the test shall be discontinued at this point.

In cases where the interface between more-or-less clear oil and the emulsion is not a clear, straight horizontal line, the position of such a line is carefully estimated to the nearest 0.5 cc.

If, at the end of 20 minutes, there is not a well-defined horizontal interface, then, and then only, the contents of the oil container may be stirred for two seconds with a clean glass rod to obtain a better line of demarcation.

Interpretation.

12. In interpreting results, the assumption shall be made that the rate of emulsion separation is directly proportional to the Resistance to Emulsification of the tested oil. In order to establish a basis for comparison, a definite value of 60 shall be assigned to an oil which, under conditions of the test, separates completely from emulsion in one minute. This is equivalent to an emulsion separation rate of 0.33 cc. per second or 20 cc. per minute.

NOTE.—The expansion of the oil at the higher temperature is disregarded, so when the separation of the oil is complete, as regards the interface between the oil and emulsion layers, the volume of separated oil will always be more than 20 cc. This in no way interferes with the reproducibility of results, and the time shall be recorded when 20 cc.—and not necessarily all—of the oil have separated, when this occurs in less than 20 minutes.

A.S.T.M. S.E. Number.

13. Results shall be reported, to the nearest second, of the time necessary for complete (20 cc.) oil separation. The time in seconds shall be reported as the Steam Emulsion Number, A.S.T.M. Method (S.E. No. ....). In cases where the required volume shall not have separated in 20 minutes, the oil shall be reported as having an S.E. Number, A.S.T.M. Method, of 1200 plus. If a duplicate determination varies by more than 10 per cent, a third test shall be made and the average of the three tests reported.

NOTE.—Oils which emulsify separate into three layers without exception: top, clear or turbid oil; middle, lacy or creamy emulsion; bottom, clear or milky water. The S.E. Number is derived from the top layer.

STANDARD METHOD OF TEST FOR SULFUR IN PETROLEUM OILS HEAVIER THAN ILLUMINATING OIL[1]

Serial Designation: D 129 – 27

This method is issued under the fixed designation D 129; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED, 1927.

APPARATUS

Oxygen Bomb.

1. The oxygen bomb shall have a capacity of not less than 300 cc., and shall be of a design or construction such that no leaks shall occur at any pressure or temperature generated during the test and such that when the bomb is open liquid contents can be easily and completely drained. The inner surfaces shall be of materials that are chemically and physically resistant to the process or products of combustion. The gaskets, insulating materials, etc., shall be, as far as possible, physically and chemically resistant and in no event shall they undergo any reaction which would increase or decrease the sulfur content of the bomb liquors.

Oil Cup.

2. The oil cup shall be of platinum, glazed silica, or other suitable material, with a capacity of not less than 2.5 cc. nor more than 5.0 cc.

Fuse Wire.

3. If a platinum oil cup is used, the fuse wire shall be of platinum; if a glazed silica oil cup is used, the fuse wire may be of either platinum or iron. No. 35 B. & S. gage is a convenient size.

Reagents.

4. (*a*) The distilled water and all reagents should be sulfur free, but in such cases where it is necessary to employ reagents not sulfur free, blanks shall be run and the figures thus obtained used to correct the results of actual determinations.

(*b*) The barium chloride solution shall contain 100 g. of $BaCL_2 \cdot 2H_2O$ per liter.

PROCEDURE

Procedure.

5. (*a*) Twenty cubic centimeters of distilled water shall be placed in the bottom of the bomb. From 0.6 to 0.8 g. of the oil to be tested

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

shall be placed in the weighed oil cup and the weight of this charge shall be determined to an accuracy of at least ±0.002 g. The cup shall be placed in the proper position in the bomb, the ignition mechanism arranged and the bomb closed. Oxygen shall be admitted slowly until a pressure is reached as indicated by the following table:

CAPACITY OF BOMB, CC.	MINIMUM GAGE PRESSURE, ATMOSPHERES.
300 to 350	40
350 to 400	35
400 to 450	30
450 to 500	27.5
Above 500	25

The leads from the firing circuit shall be attached, the bomb placed in a bucket of cold water, and ignited. The bomb shall be allowed to stand in the water for 10 minutes and shall then be removed. The valve of the bomb shall be opened, allowing the gas to escape at an approximately even rate so that the pressure is reduced to atmospheric in not less than 1 minute. The bomb shall be opened, and all parts of its interior, including the oil cup, rinsed with a fine jet of distilled water. All washings, which should not amount to more than 350 cc., shall be collected in a beaker. Particular care should be taken not to lose, by splashing, or otherwise, any of the liquid contents of the bomb. The washings shall be filtered through a washed "qualitative" filter paper. The filter shall be washed thoroughly. Two cubic centimeters of concentrated HCl and 10 cc. of saturated bromine water shall be added to the filtrate. The solution shall be evaporated to about 75 cc. on a steam bath or hot plate. Ten cubic centimeters of hot barium chloride solution shall be added in a fine stream or drop-wise to the hot solution, stirring during the addition and for two minutes afterward. The solution shall be allowed to stand over night, or shall be kept hot for one hour on the steam bath or hot plate, allowing the precipitate to settle for another hour, while cooling. The supernatant liquid shall be filtered through an "ashless quantitative" filter paper, the precipitate washed with water, first by decantation, then on the filter, till free from chloride. The paper and precipitate shall be transferred to a suitable weighed crucible, dried at low heat till moisture is evaporated, the paper charred (without flaming), and finally ignited at a good red heat till the precipitate is just burned white. A satisfactory means of accomplishing these operations is to place the crucible containing the wet filter paper in a cold electric muffle furnace and to turn on the current. Drying, charring, and ignition will usually occur at the desired rate.

(*b*) After ignition is complete, the crucible shall be allowed to cool to room temperature, and weighed. The use of a desiccator is not recommended.

(*c*) From the increase in weight of the crucible the percentage of sulfur shall be calculated from the formula:

$$\text{Percentage of sulfur} = \frac{\text{grams of } BaSO_4 \times 13.734}{\text{grams of oil used.}}$$

ACCURACY

6. Duplicate determinations should agree to ±2 per cent on the basis of the sulfur found. **Accuracy.**

STANDARD METHOD OF TEST
FOR
THERMAL VALUE OF FUEL OIL[1]

Serial Designation: D 240 – 27

This method is issued under the fixed designation D 240; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1926; ADOPTED IN AMENDED FORM, 1927.

APPARATUS

Combustion Bombs.

1. The Atwater, Davis, Emerson, Mahler, Parr, Peters, Williams or similar bombs may be used. The bomb shall have an inner surface of platinum, gold, porcelain enamel or other material which is not attacked by nitric and sulfuric acids, or other products of combustion.

Calorimeter Jacket.

2. The calorimeter shall be provided with a vacuum jacket or with a water jacket having a cover to protect the calorimeter from air currents. In case a water jacket is used, it shall be kept filled with water within 2 or 3° C. of the temperature of the room (except in calorimeters which are totally submerged, where the jacket temperature is controlled by a thermostat) and this water should be stirred continuously by some mechanical stirring device.

Stirring of the Calorimeter Water.

3. The water in the calorimeter shall be stirred sufficiently well to give consistent thermometer readings while the temperature is rising rapidly. The speed of stirring should be kept constant. A motor-driven screw or turbine stirrer is recommended and the speed should not be excessive. This may be determined by adjusting the temperature of the calorimeter to equality with that of the jacket and allowing the stirrer to run continuously for 10 minutes. If the temperature of the calorimeter rises more than about 0.01° C. in this length of time, the rate of stirring is excessive. Accurate results cannot be obtained when too much energy is supplied by the stirring device or when the rate of stirring is irregular. The portion of the stirring device immersed in the calorimeter should be separated from that outside by non-conducting material, such as hard rubber, to prevent conduction of heat from the motor or outside air.

Thermometers.

4. The thermometers used shall have been certified by a government testing bureau and shall be used with the corrections given on

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

the certificate. This shall also apply to electrical resistance or thermoelectric thermometers. Correction shall also be made for the temperature of the emergent stem of all mercurial thermometers, and for the "setting" of Beckmann thermometers. For accurate work, either Beckmann or special calorimetric thermometers, graduated to 0.01 or 0.02° C., are required. Such thermometers should be tapped lightly just before each reading to avoid errors caused by the sticking of the mercury meniscus, particularly when the temperature is falling. A convenient method is to mount a small electric buzzer directly on the top of the thermometer and connect it with a dry cell and a push button. The button should be pressed for a few seconds immediately before each reading.

Oxygen.

5. The oxygen used for combustions shall be free from combustible material. The bomb when filled should contain at least 5 per cent of nitrogen to insure complete oxidation of the sulfur. The total amount of oxygen contained in the bomb for combustion shall be not less than 5 g. per gram of fuel oil. But the combustion must be complete, as shown by the absence of any sooty deposit on opening the bomb after firing.

Firing Wire.

6. The fuel oil in the bomb may be ignited by means of either iron or platinum wire. If iron wire is used, it should be of about No. 34 B. & S. gage and not more than 10 cm. (preferably 5 cm.) should be used at a time. A correction of 1600 calories per gram weight of iron wire burned shall be subtracted from the observed number of calories.

Standardization.

7. The water equivalent of a calorimeter can best be determined by the use of the standard combustion samples supplied by the U. S. Bureau of Standards. The required water equivalent is equal to the weight of the sample multiplied by its heat of combustion per gram and divided by the corrected rise in temperature.

The calorimeter shall be standardized by the combustion of standard samples supplied by the Bureau of Standards, and used according to the directions given in the certificates which accompany them. A standardization shall consist of a series of not less than five combustions of each of two of the standard materials which are, sucrose, naphthalene and benzoic acid. The conditions as to the amount of water, oxygen, firing wire, method of correcting for radiation, etc., under which these combustions are made, shall be the same as for fuel oil combustions. In the case of any disagreement between contracting parties a check standardization may consist of two or more combustions of standardizing samples.

PROCEDURE

Preparation of Sample.

8. Approximately enough oil shall be taken to give a thermometer rise of from 3 to 4° C. After weighing, the sample should preferably be immediately placed in the bomb and this closed. This procedure is necessary to avoid sublimation in the use of naphthalene for standardization.

Preparation of the Bomb.

9. The firing wire, if iron, shall be measured and coiled in a small spiral and connected between the platinum terminals, using, if necessary, a piece of platinum wire somewhat heavier than the iron wire to make the connection. The platinum and the iron shall both be clean. About 0.5 cc. of water should be placed in the bottom of the bomb to saturate with moisture the oxygen used for combustion. When the crucible is put in place in the bomb, the firing wire should touch the fuel oil or briquet of standard material. For the combustion of standardizing samples, iron wire is preferable to platinum.

Filling the Bomb with Oxygen.

10. Oxygen from the supply tank shall be admitted slowly, to avoid blowing the oil from the crucible, until a pressure is reached as indicated by the following table:

CAPACITY OF BOMB, CC.	MINIMUM GAGE PRESSURE, ATMOSPHERES
300 to 350	40
350 to 400	35
400 to 450	30
450 to 500	27.5
Above 500	25

Calorimeter Water.

11. The calorimeter shall be filled with the required amount of distilled water, depending upon the type of calorimeter. The amount may be determined either by measurement in a standardized flask or by weighing. The amount shall be kept the same as that used in the standardization of the apparatus.

Temperature Adjustments.

12. The initial temperature in the calorimeter shall be so adjusted that the final temperature, after the combustion, will not be more than 1° C., preferably about 0.5° C., above that of the jacket. Under these conditions, the total correction for heat gained from or lost to the surroundings will be small when the rise of temperature is 3 or 4° C. and the effect of evaporation will also be small.

Firing Current.

13. The electric current used for firing the charge shall be obtained from storage or dry cells having an electromotive force of not more than 12 volts. A higher voltage is liable to cause an arc between the firing terminals, introducing additional heat which cannot be measured with certainty. The circuit should be closed by means of a switch which should remain closed for not more than 2 seconds. When

possible, it is recommended that an ammeter be used in the firing circuit to indicate when the firing wire has burned out.

Method of Making an Observation.

14. The bomb, when ready for firing, shall be placed in the calorimeter, the firing wires connected, the cover put in place and the stirrer and thermometer so placed as not to be in contact with the bomb or container. The stirrer shall then be started and after the thermometer reading has become steady, not less than 2 minutes after the stirrer is started, the temperatures shall be read at 1-minute intervals for 5 minutes and the charge then fired, the exact time of firing being noted. Observations of temperature shall then be made at intervals depending upon the method to be used for computing the cooling correction. When the temperature has reached its maximum and is falling uniformly, a series of thermometer readings shall be taken at 1-minute intervals for 5 minutes to determine the final cooling rate.

Titration.

15. After a combustion and after allowing the gas to escape, the bomb shall be opened and the inside examined for traces of unburned material or sooty deposit. If these are found, the observations shall be discarded. If the combustion appears complete, the bomb shall be rinsed out thoroughly and the washings titrated with a standard alkali solution (1 cc. = 0.02173 g. HNO_3 = 5 calories) using methyl-orange or methyl-red indicator to determine the amount of acid formed. A correction of 230 calories per gram of HNO_3 should be subtracted from the total heat observed. An additional correction of 1300 calories per gram of sulfur in the oil should be made for the excess of difference in heats of formation of SO_2 and aqueous H_2SO_4 over the heat of formation of aqueous HNO_3.

COMPUTATION OF RESULTS

Computation of Results.

16. The following method of computation is recommended to take the place of the Pfaundler or other similar formulas for computing the cooling correction (radiation correction):

Observe (1) the rate of rise, r_1, of the calorimeter temperature in degrees per minute for 5 minutes before firing; (2) the time, a, at which the last temperature reading is made immediately before firing; (3) the time, b, when the rise of temperature has reached six-tenths of its total amount (this point can generally be determined by adding to the temperature observed before firing, 60 per cent of the expected temperature rise, and noting the time at which this point is reached); (4) the time, c, of a thermometer reading taken when the temperature change has become uniform some 5 minutes after firing; (5) the final rate of cooling, r_2, in degrees per minute for 5 minutes.

Multiply the rate r_1 by the time $b - a$ in minutes and tenths of a minute, and add to this product (subtracted if the temperature was falling at the time a) to the thermometer reading taken at the time a. Multiply the rate r_2 by the time $c - b$ and add this product (subtracted if the temperature was rising at the time c and later) to the thermometer reading taken at the time c. The difference of the two thermometer readings thus corrected, provided the corrections from the certificate have already been applied, gives the total rise of temperature due to the combustion. This, multiplied by the water equivalent of the calorimeter, gives the total amount of heat liberated. Divide this result, corrected for the heats of formation of HNO_3 and H_2SO_4 observed and for the heat of combustion of the firing wire, when that is included, by the weight of the charge to find the heat of combustion in calories per gram. Calories per gram multiplied by 1.8 give the British thermal units per pound.

Duplicate Determinations.

17. The permissible differences in duplicate determinations are as follows:

Same analyst	0.3 per cent
Different analysts	0.5 per cent

18. In practice, the time $b - a$ will be found so nearly constant for a given calorimeter with the usual amounts of fuel that b need be determined only occasionally.

The results shall be reduced to calories per gram or British thermal units per pound of oil.

STANDARD METHODS OF TEST
FOR
VISCOSITY OF PETROLEUM PRODUCTS AND LUBRICANTS[1]

Serial Designation: D 88 – 26

These methods are issued under the fixed designation D 88; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1926.

Choice of Instrument.

1. (*a*) Viscosity shall be determined by means of the Saybolt Universal or Saybolt Furol Viscosimeter.

(*b*) In general, the Saybolt Universal Viscosimeter shall be used for lubricants and the Saybolt Furol Viscosimeter for fuel oils and other oils of similar viscosity.

TABLE I.—DIMENSIONS OF OIL TUBES.

Dimensions	Saybolt Universal Viscosimeter			Saybolt Furol Viscosimeter		
	Minimum, cm.	Normal, cm.	Maximum, cm.	Minimum, cm.	Normal, cm.	Maximum, cm.
Inside diameter of outlet tube	0.1750	0.1765	0.1780	0.313	0.315	0.317
Outside diameter of outlet tube at lower end.	0.28	0.30	0.32	0.40	0.43	0.46
Length of outlet tube[1]	1.215	1.225	1.235	1.215	1.225	1.235
Height of overflow rim above bottom of outlet tube[1]	12.40	12.50	12.60	12.40	12.50	12.60
Outside diameter of overflow rim, at the top[1,b]	[a]	....	3.30	[a]	....	3.30
Diameter of container[1]	2.955	2.975	2.995	2.955	2.975	2.995
Depth of cylindrical part of container[1]	8.8	....	....	8.8	....	....
Diameter of container between bottom of cylindrical part of container and top of outlet tube[1]	0.9	....	....	0.9	....	....

[1] This dimension is identical in the Saybolt Universal and the Saybolt Furol instruments.
[a] The minimum value shall preferably be not less than 3.2 cm.
[b] The section of overflow rim shall be bounded by straight lines, except that a fillet is permissible at the junction with the bottom of the gallery.

(*c*) The Saybolt Universal Viscosimeter shall not be used for times of flow less than 32 seconds.

APPARATUS

Viscosimeters.

2. (*a*) The Saybolt viscosimeters (see Fig. 1) are made entirely of metal. The oil tube, *A*, is fitted at the top with an overflow cup, *B*, and the tube is surrounded by a bath. At the bottom of the oil tube is a small outlet tube through which the oil to be tested flows into a receiving flask, Fig. 2, whose capacity at 20° C. (68° F.) to a mark on its neck is 60 ±0.15 cc. The outlet tube is of hard and

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

corrosive-resistant metal such as stainless steel, monel metal, etc. The inside diameter of neck of flask, at the level where the graduation mark is placed, is 0.8 to 1.1 cm. The lower end of the outlet tube is enclosed by a larger tube, which when stoppered by a cork, *C*, acts as a closed air chamber and prevents the flow of oil through the outlet tube until the cork is removed and the test started. A looped string

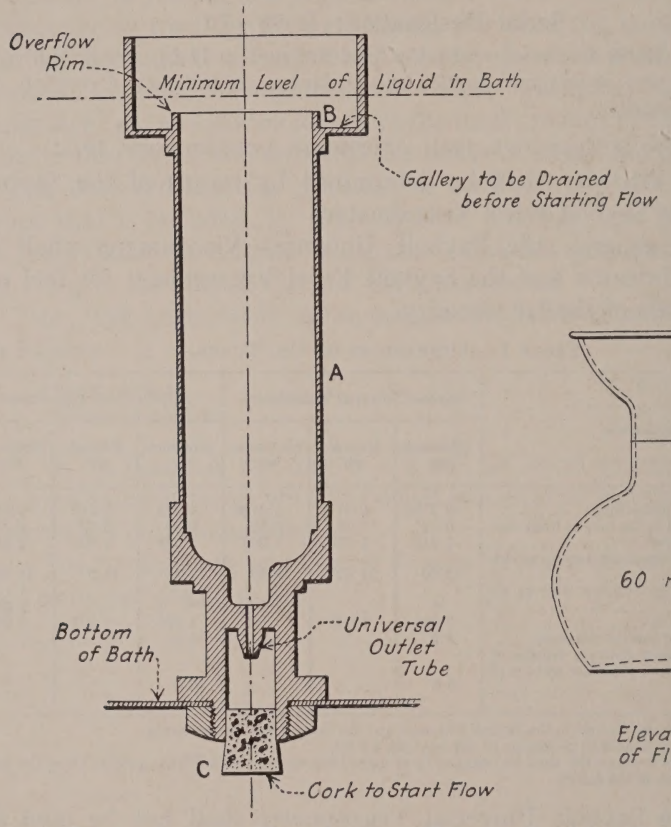


FIG. 1.—Sectional View of Standard Oil Tube.

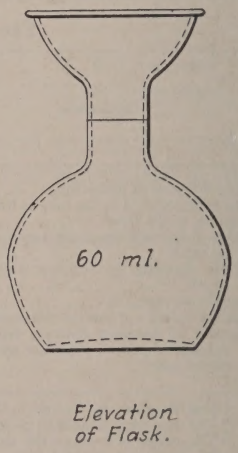


FIG. 2.—Receiving Flask. (This type of flask is recommended as convenient and durable.)

may be attached to the lower end of the cork as an aid to its rapid removal. The temperatures in the oil tube and in the bath are shown by thermometers. The bath may be heated by any suitable means. The oil tube shall be thoroughly cleaned, and all oil entering the oil tube shall be strained through a 100-mesh wire strainer. A stop watch shall be used for taking the time of flow of the oil and a pipette shall be used for draining the overflow cup.

(*b*) The oil tubes, which may be standardized by the U. S. Bureau of Standards, shall conform to the dimensions given in Table I. The time of flow shall be within ±1 per cent of the time as obtained with the Bureau of Standards' master tube.[1] **Oil Tube.**

(*c*) The bath and oil tube thermometers shall conform to the following requirements. They cover two sets of 4 thermometers each, one set being graduated in Fahrenheit degrees and the other set in Centigrade degrees, the ranges being chosen to include the temperatures commonly used in testing. **Thermometers.**

TYPE: Etched stem, glass.
LIQUID: Mercury.
RANGES AND SUBDIVISIONS:

Range	66– 80° F.,	subdivision in	0.2° F.	for tests at	77° F.
"	94–108° F.,	"	0.2° F.	"	100° F.
"	120–134° F.,	"	0.2° F.	"	122 and 130° F.
"	204–218° F.,	"	0.2° F.	"	210° F.

or

Range	19–27° C.,	subdivision in	0.1° C.	for tests at	25° C.
"	34–42° C.,	"	0.1° C.	"	40° C.
"	49–57° C.,	"	0.1° C.	"	50° C.
"	95–103° C.,	"	0.1° C.	"	100° C.

TOTAL LENGTH: 252 to 256 mm. (9.92 to 10.08 in.).
STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6 to 7 mm. (0.236 to 0.275 in.). The stem shall preferably be made with an enlargement not less than 4 nor more than 7 mm. (0.16 to 0.28 in.) in length, having a diameter 2 to 3 mm. (0.08 to 0.12 in.) greater than that of the stem, the bottom of the enlargement being 114 mm. (4.5 in.) above the bottom of the bulb.
BULB: Corning normal or equally suitable thermometric glass.
Length, 25 to 35 mm. (1.0 to 1.4 in.).
Diameter, not less than 5 mm. (0.197 in.) and not greater than that of stem.
DISTANCE TO LOWEST SPECIFIED GRADUATION FROM BOTTOM OF BULB: 135 to 150 mm. (5.3 to 5.9 in.).
DISTANCE TO HIGHEST SPECIFIED GRADUATION FROM TOP OF STEM: 20 to 35 mm. (0.8 to 1.4 in.).
CONTRACTION CHAMBER: To be of long narrow type, top to be not more than 60 mm. (2.36 in.) above bottom of bulb, mercury to stand in contraction chamber at 32° F. (0° C.).
EXPANSION CHAMBER: To permit heating the thermometer 90° F. (50° C.) above highest temperature on scale, and in all cases to permit heating to 212° F. (100° C.).
FILLING ABOVE MERCURY: Nitrogen gas.
TOP FINISH: Glass ring.
GRADUATION: All lines, figures and letters clear cut and distinct. The first and each succeeding 1° F. or 0.5° C. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 2° F. or 1° C.
IMMERSION: Total.

[1] This tube represents the average mechanically perfect tube, based on the A.S.T.M. specifications, and has been approved by the American Petroleum Institute Committee on Viscosity Standards.

SPECIAL MARKING: "A.S.T.M." Viscosity test points to be numbered in full figures and in red, other graduations and figures in black. A serial number and the manufacturer's name or trade mark shall be etched on the stem.

SCALE ERROR: The error at any point of the scale shall not exceed 0.2° F. (0.1° C.).

STANDARDIZATION: The thermometers are to be standardized for the condition of total immersion. Correction for emergent stem shall not be applied.

CASE: Each thermometer shall be supplied in a suitable case on which shall appear the marking, "A.S.T.M., Saybolt Viscosimeter Thermometer" and the range.

TEMPERATURE OF TESTING

Temperature.

3. (*a*) With the Saybolt Universal viscosimeter, determinations shall be made at 100° F. (37.8° C.), 130° F. (54.4° C.) or 210° F. (98.9° C.).

(*b*) With the Saybolt Furol viscosimeter, determinations shall be made at 122° F. (50° C.).

(*c*) Viscosities shall be expressed as ..... seconds, Saybolt Universal (or Saybolt Furol), being the time in seconds for the delivery of 60 cc. of oil.

(*d*) Fuel oils and other oils of similar viscosity showing a time of less than 25 seconds, Saybolt Furol, at 122° F., shall be tested on the Saybolt Universal at 122° F. Oil showing a time of less than 32 seconds, Saybolt Universal, at 122° F., shall be measured in the Saybolt Universal at 100° F. (37.8° C.). These methods of test do not apply to fuels having a viscosity at 100° F. of less than 32 seconds Saybolt Universal, which are not considered to be fuel oils.

PROCEDURE

Procedure.

4. In tests at 100 and 130° F. (37.8 and 54.4° C.) the bath temperature throughout the test shall not vary more than ±0.1° F. (0.06° C.) from the predetermined temperature which will maintain thermal equilibrium as long as the oil is well stirred with the thermometer. In tests at 210° F. (98.9° C.) a variation of ±0.2° F. (0.11° C.) is permitted.

Any construction of bath may be employed provided the bath temperature necessary to maintain thermal equilibrium (while the oil in the oil tube is well stirred by the oil tube thermometer) is not in excess of 100.25, 122.35, 130.50 and 212.00° F. (37.9, 50.2, 54.7 and 100.00° C.), respectively, for the standard temperatures previously mentioned. The level of the bath liquid shall not be lower than 0.5 cm. above the overflow rim of the oil tube. In tests at 210° F. (98.9° C.) the water bath shall be vigorously stirred, and may be heated by the direct injection of steam near the bottom of the bath. A bath of other suitable liquid may be used provided it is properly heated and

stirred.[1] The heating and stirring of the bath may be accomplished by any suitable means, provided the source of heat is not less than the following distances from any part of the oil tube: 2 in. (5 cm.) with an external heater, $1\frac{1}{4}$ in. (3 cm.) with an immersion heater. Viscosity determinations shall be made in a room free from draughts and from rapid changes in temperature. The room temperature shall be between 68 and 86° F. (20 and 30° C.).[2]

All oil introduced into the oil tube, either for cleaning or for test, shall first be passed through the strainer.

To make the test, heat the oil to the necessary temperature and clean out the oil tube. Pour some of the oil to be tested through the cleaned tube. Insert the cork stopper into the lower end of the air chamber at the bottom of the oil tube, sufficiently to prevent the escape of air, but not to touch the small outlet tube.

Heat the oil to be tested, outside the viscosimeter, to slightly but not more than 3° F. (1.7° C.) above the temperature at which the viscosity is to be determined, and pour it into the oil tube until it ceases to overflow into the overflow cup. By means of the oil tube thermometer, keep the oil in the oil tube well stirred, and also stir well the liquid in the bath. It is extremely important that the temperature of the bath be maintained constant during the entire time consumed in making the test. After the temperature of the bath and of the oil in the oil tube have been constant for one minute at the desired temperatures, withdraw the oil tube thermometer, quickly remove the surplus oil from the overflow cup by means of a pipette[3] so that the level of the oil in the overflow cup is below the level of the oil in the oil tube proper; place the 60-cc. flask, Fig. 2, in position so that the stream of oil from the outlet tube will strike the neck of the flask so as to avoid foam. Snap the cork from its position, and at the same instant start the stop-watch. Stir the liquid in the bath during the run and carefully maintain it at the previously determined proper temperature. Stop the watch when the bottom of the meniscus of the oil reaches the mark on the neck of the receiving flask.

[1] For example, the bath liquid may be water, to which glycerin or salt has been added. In ordinary routine testing it is frequently desirable to employ oil as a bath medium. This is allowable provided the temperature of the oil bath is adjusted so that the necessary condition of thermal equilibrium is maintained. It is usually necessary to maintain the oil bath at slightly higher temperatures than are necessary when water is the bath medium. Oil bath temperatures will need to be from 0.1 to 0.2° F. (0.06 to 0.11° C.) higher for tests at 100° F. (37.8° C.) and 1.5 to 2.0° F. (0.85 to 1.11° C.) higher for tests at 210° F. (98.9° C.) than the corresponding water bath temperatures.

[2] These limits are necessary for extreme accuracy in standardization and referee tests, but for routine purposes the use of higher temperatures up to 100° F. (37.8° C.) will not ordinarily cause an error of more than 1 per cent.

[3] It is recommended that the tip of the pipette should be about 2 mm. inside, and about 3 mm. outside diameter, and great care should be exercised to avoid touching the overflow rim while draining the overflow cup.

The time in seconds for the delivery of 60 cc. of oil is the Saybolt Universal (or Saybolt Furol) viscosity of the oil at the temperature at which the test is made.

With proper attention to details of methods of procedure, duplicate results should not differ from each other by more than 1 per cent.

STANDARD METHOD OF TEST
FOR
WATER IN PETROLEUM PRODUCTS AND OTHER BITUMINOUS MATERIALS[1]

Serial Designation: D 95 – 27

This method is issued under the fixed designation D 95; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1924; REVISED, 1927.

Scope.

1. This method of test determines water existing in a sample of bituminous material by distilling the sample with a volatile solvent. This method is suitable for a variety of materials but is especially applicable to petroleum, fuel oil, road oil, coal tar, water-gas tar, coke-oven tar and other petroleum products or bituminous materials.

APPARATUS

General

2. The apparatus shall consist of a metal still or glass flask, heated by suitable means and provided with a reflux condenser discharging into a trap connected to the still or flask. The trap serves to collect and measure the condensed water and to return the solvent to the still.

The type of distilling apparatus used is not an essential feature of this method, but glass has been generally used for petroleum products and the metal still for road materials and tars.

Metal Still.

3. (*a*) The metal still (Fig. 1(*a*)) shall be a vertical cylindrical vessel, preferably of copper, having a faced flange at the top to which the head is tightly attached by means of a clamp. The head shall be of metal, preferably of brass or copper, and be provided with a tubulation 1 in. in inside diameter.

Glass Still.

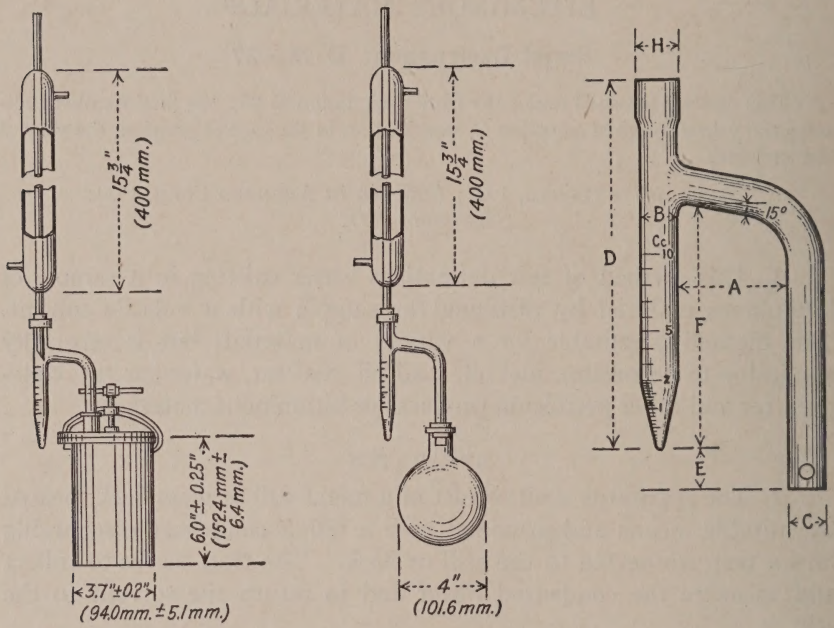
(*b*) The glass flask (Fig. 1(*b*)) shall be of the short neck, round-bottom type, made of well-annealed glass, having an approximate capacity of 500 cc.

Heat Source.

4. The burner used with the metal still shall be a ring gas burner 4 in. (100 mm.) in inside diameter. With the glass flask, an ordinary gas burner or electric heater may be used as the source of heat.

[1] Under the standardization procedure of the Society, this method is under the joint jurisdiction of the A.S.T.M. Committees D-2 on Petroleum Products and Lubricants and D-4 on Road and Paving Materials.

Condenser. 5 The condenser shall be of the water-cooled, reflux, glass-tube type, having a condenser jacket not less than 400 mm. ($15\frac{3}{4}$ in.) in length with an inner tube 12 to 16 mm. ($\frac{1}{2}$ to $\frac{5}{8}$ in.) in diameter. The end of the condenser to be inserted in the trap shall be ground off at an angle of 60 deg.



A	45 to 55 mm.	$1\frac{3}{4}$" to $2\frac{1}{8}$"
B (Inside Diameter)	14 " 16 mm.	$\frac{9}{16}$" " $\frac{5}{8}$"
C (Inside Diameter)	12 " 16 mm.	$\frac{1}{2}$" " $\frac{5}{8}$"
D	150 " 170 mm.	6" " $6\frac{3}{4}$"
E	25 " 38 mm.	1" " $1\frac{1}{2}$"
F	100 " 108 mm.	4" " $4\frac{1}{4}$"
H (Inside Diameter)	18 " 19 mm.	$\frac{11}{16}$" " $\frac{3}{4}$"

(*a*) (*b*) (*c*)

FIG. 1.

Trap. 6. The trap shall be made of well-annealed glass constructed in accordance with Fig. 1 (*c*) and shall be graduated as shown from 0 to 10 cc. in 0.1 cc. divisions. The error of any indicated capacity shall not be greater than 0.05 cc.

The outside diameters should be preferably 2.5 to 3.5 mm. ($\frac{3}{32}$ to $\frac{1}{8}$ in.) greater than the inside diameters specified.

Solvent. 7. (*a*) The solvent used when testing petroleum products or bituminous materials derived from petroleum shall be gasoline free from water

and shall conform to the following distillation requirements, determined in accordance with the Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (Serial Designation: D 86) of the American Society for Testing Materials:[1]

5 per cent shall distill at a temperature not below 194° F. (90° C.) nor above 212° F. (100° C.)
90 per cent shall distill below 410° F. (210° C.)

(*b*) The solvent used when testing bituminous materials derived from coal tar, water-gas tar, etc., shall be a coal-tar naphtha or a light oil and shall conform to the following distillation requirements, determined in accordance with the Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (Serial Designation: D 86) of the American Society for Testing Materials:[1]

98 per cent shall distill between 248° F. (120° C.) and 482° F. (250° C.)

SAMPLE

Sample.

8. The sample shall be thoroughly representative of the material to be tested and the portion of the sample used for the test shall be thoroughly representative of the sample itself. Deviation from this requirement shall not be permitted.

NOTE.—The difficulties in obtaining proper representative samples for this determination are unusually great so that the importance of sampling cannot be too strongly emphasized.

PROCEDURE

Procedure.

9. When the sample to be tested contains less than 10 per cent of water, exactly 100 cc. of the material to be tested shall be placed into the still or flask and thoroughly mixed with an equal volume of solvent by swirling, proper care being taken to avoid any loss of material. If the material is measured by volume, an accurate 100-cc. graduated cylinder shall be used and the contents transferred to the still by rinsing with one 50-cc. portion of solvent followed by two successive 25-cc. portions of solvent, the cylinder being allowed to drain each time. When the sample to be tested contains more than 10 per cent of water, the volume of material used shall be decreased to that which will yield somewhat less than 10 cc. of water.

NOTE.—In special cases where the water content exceeds 10 per cent and it is not desirable to reduce the size of the sample to that which will yield somewhat less than 10 cc. of water, a distilling tube receiver graduated from 0 to 25 cc. may be used. This tube shall be graduated from 0 to 2 cc. in 0.1 cc., from 2 to 5 cc. in 0.2 cc. and from 5 to 25 cc. in 0.5 cc.

[1] See p. 378.

The connections between the still or flask, trap and condenser shall be made by means of tight-fitting corks as shown in Figs. 1 (*a*) and (*b*). When the metal still is used, a heavy paper gasket moistened with the solvent shall be inserted between the lid and flange before attaching the clamp. A loose cotton plug shall be inserted in the top of the condenser tube to prevent condensation of atmospheric moisture in the condenser tube.

Heat shall then be applied and so regulated that the condensed distillate falls from the end of the condenser at the rate of from 2 to 5 drops per second. The ring burner used with the metal still should be placed about 3 in. above the bottom of the still at the beginning of the distillation and gradually lowered as the distillation proceeds.

The distillation shall be continued at the specified rate until no water is visible on any part of the apparatus except at the bottom of the tray. This operation usually requires less than an hour. A persistent ring of condensed water in the condenser tube shall be removed by increasing the rate of distillation for a few minutes.

Calculation of Results.

10. The volume of condensed water measured in the trap at room temperature multiplied by 100 and divided by the volume of the sample used shall be the percentage of water and shall be reported as "..... per cent water by volume, A.S.T.M. method."

ACCURACY

Accuracy.

11. The accuracy to be expected with this method is that duplicate determinations of water should not differ from each other by more than one division on the trap.

STANDARD METHOD OF TEST
FOR
WATER AND SEDIMENT IN PETROLEUM PRODUCTS, BY MEANS OF CENTRIFUGE[1]

Serial Designation: D 96 – 24

This method is issued under the fixed designation D 96; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1924.

Scope.

1. This method may be used for crude mineral oils and fuel oils. A centrifuge method for "Water and Sediment" is not entirely satisfactory because the amount of water obtained is nearly always lower than the actual water content. Nevertheless, on account of the wide use of the centrifuge for this purpose, it is desirable that the method of making the determination be standardized as far as possible. It must be clearly understood that the reading of the centrifuge tube includes both the sediment and the precipitated water. Accurate determination of water content, if desired, should be made in accordance with the Standard Method of Test for Water in Petroleum Products and other Bituminous Materials (Serial Designation: D 95) of the American Society for Testing Materials.[2]

Sample.

2. The sample shall be thoroughly representative of the material in question and the portion used for the test shall be thoroughly representative of the sample itself. Deviation from this rule shall not be permitted. The difficulties in obtaining proper representative samples for this determination are unusually great; hence, the importance of sampling cannot be too strongly emphasized.

APPARATUS.

Centrifuge.

3. The centrifuge shall be capable of whirling at the required speed at least two 100-cc. centrifuge tubes filled with water. It shall be of sound design and rugged construction so that it may be operated without danger. The tube carriers shall be so designed that the glass centrifuge tubes may be cushioned with water, rubber or other suitable material. The tube holders shall be surrounded during the

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

[2] See p. 433.

operation by a suitable metal shield or case, strong enough to eliminate danger if any breakage occurs.

The preferred form of centrifuge shall have a diameter of swing (tip to tip of whirling tubes) of 15 to 17 in. and a speed of at least

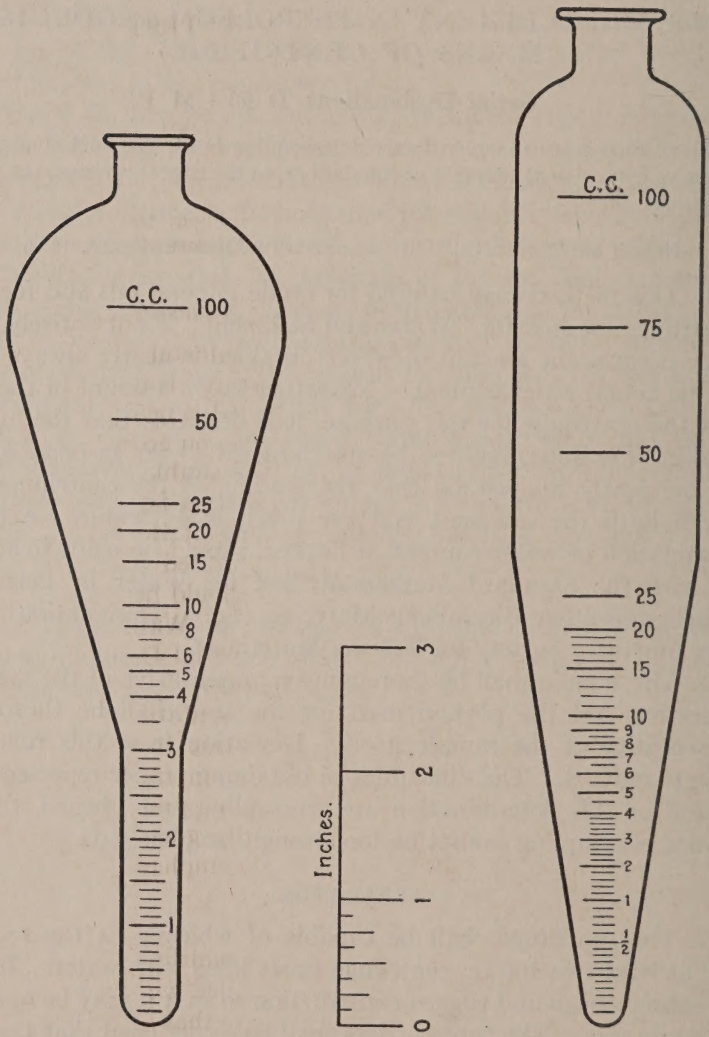


FIG. 1.

1500 r.p.m. or the equivalent. If the available centrifuge has a diameter of swing varying from these limits, it shall be run at the proper speed to give the same centrifugal force at the tips of the

tubes as that obtained with the preferred form of centrifuge. The proper speed shall be calculated from the following formula in which d represents diameter of swing (tip to tip of whirling tubes) of the centrifuge used:

$$\text{R.p.m.} = 1500 \sqrt{\frac{16}{d}}$$

Centrifuge Tubes.

4. The centrifuge tubes, A.S.T.M. type, shall be made of suitable glass and thoroughly annealed. The total capacity shall be about 125 cc. and the mouth shall be suitably constricted for closing with a cork. The graduations shall be clear and distinct, reading upward from the bottom of the tube as follows:

RANGE.	SCALE DIVISIONS.	LIMIT OF ERROR.	NUMBERED.
0 - 3 cc.	0.1 cc.	0.05 cc.	1, 2, 3 cc.
3 - 5 "	0.5 "	0.2 "	4, 5 "
5 - 10 "	1.0 "	0.5 "	6, 8, 10 "
10 - 25 "	5.0 "	1.0 "	15, 20, 25 "
50 - 100 "	50.0 "	1.0 "	50, 100 "

The shape is optional provided it does not conflict with the other requirements. Satisfactory types are shown in Fig. 1.

Bath.

5. The water or oil bath shall be of sufficient depth for immersing the centrifuge tubes in a vertical position to the 100-cc. mark. Means shall be provided for heating this bath to 120° F.

PROCEDURE.

Procedure.

6. (*a*) Exactly 50 cc. of 90-per-cent benzol shall be measured into each of two centrifuge tubes and exactly 50 cc. of the oil to be tested shall then be added to each. The centrifuge tubes shall be tightly stoppered and shall be shaken vigorously until the contents are thoroughly mixed. The temperature of the bath shall be maintained at 120° F. and the centrifuge tubes shall be immersed therein to the 100-cc. mark for 10 minutes.

(*b*) The two centrifuge tubes shall then be placed in the centrifuge on opposite sides and shall be whirled at a rate of 1400 to 1500 r.p.m. or the equivalent for 10 minutes. The combined volume of water and sediment at the bottom of each tube shall be read and recorded, estimating to 0.1 cc. if necessary. The centrifuge tubes shall then be replaced in the centrifuge, again whirled for 10 minutes as before and removed for reading the volume of water and sediment as before. This operation shall be repeated until the combined volume of water and sediment in each tube remains constant for three consecutive readings. In general, not more than four whirlings will be required.

Water and Sediment.

7. The combined total volume of water and sediment shall be read on each tube, estimating to 0.1 cc. if necessary. The sum of the two readings shall be recorded as percentage of water and sediment, centrifuge method.

ACCURACY.

Accuracy.

8. With care and proper attention to details, duplicate determinations of water and sediment by this method should not differ by more than 0.2 cc. provided the centrifuge tubes are accurate and readable to this degree.

STANDARD SPECIFICATIONS
FOR
MATERIALS FOR CEMENT GROUT FILLER FOR BRICK AND STONE BLOCK PAVEMENTS[1]

Serial Designation: D 57 – 20

These specifications are issued under the fixed designation D 57; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1919; ADOPTED, 1920.

These specifications were approved July 18, 1924, as "Tentative American Standard" by the American Engineering Standards Committee.

SAND FOR CEMENT GROUT FILLER

Sand.

1. The sand shall consist of clean, hard, durable stone particles, preferably siliceous, free from lumps of clay and objectionable foreign matter.

Grading.

2. (*a*) The sand shall be well graded from coarse to fine and when tested by means of laboratory sieves shall meet the following requirements:

	Per Cent.
Total passing a No. 10 sieve	100
Total passing a No. 20 sieve....not less than	80
Total passing a No. 200 sieve....not more than	5

(*b*) This test shall be made in accordance with the Standard Method of Mechanical Analysis of Sand or Other Fine Highway Material, Except Fine Aggregates Used in Cement Concrete (Serial Designation: D 7) of the American Society for Testing Materials.[2]

Mortar Strength Test.

3. (*a*) When the sand is mixed with Portland cement passing the requirements of the Standard Specifications and Tests for Portland Cement (Serial Designation: C 9) of the American Society for Testing Materials,[3] in the proportion of 1 part of cement to 3 parts of sand, by weight, according to the standard method of making 1:3 mortar briquets, the resulting

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] See p. 479. [3] See p. 23.

mortar at the age of 7 and 28 days shall have a tensile strength of at least 75 per cent of that developed in the same time by mortar of the same consistency, made of 1 part of the same cement and 3 parts of Ottawa sand.

(*b*) Preliminary acceptance samples shall be subjected to both 7 and 28-day mortar tests, and acceptance based thereon. Samples tested during the progress of the work will be accepted on the basis of the 7-day test.

(*c*) The tests for mortar strength shall be made in accordance with the A.S.T.M. Standard Specifications and Tests for Portland Cement.

NOTE.—The Portland cement used in testing the proposed sand should preferably be that used or to be used in the work.

Inspection. 4. The sand shall be subject to inspection after delivery at the place of use, prior to and during the laying of the pavement.

Sampling. 5. The sand shall be sampled for testing at the source of supply, if required. Samples shall also be taken for testing from time to time, at the place of use prior to and during the laying of the pavement, whenever desired by the engineer.

PORTLAND CEMENT FOR CEMENT GROUT FILLER

Cement. 6. Portland cement for use in cement grout filler shall conform to the requirements of the A.S.T.M. Standard Specifications and Tests for Portland Cement.

WATER FOR CEMENT GROUT FILLER

Water. 7. Clean water free from harmful amounts of oil, acid, alkali, organic or vegetable matter shall be used.

STANDARD SPECIFICATIONS

FOR

MATERIALS FOR CEMENT MORTAR BED FOR BRICK, STONE BLOCK, WOOD BLOCK, ASPHALT BLOCK AND OTHER BLOCK PAVEMENTS[1]

Serial Designation: D 58 – 24

These specifications are issued under the fixed designation D 58; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1919; ADOPTED, 1920; REVISED, 1924.

SAND FOR CEMENT MORTAR CUSHION

1. The sand shall consist of clean, hard, durable stone particles, preferably siliceous, free from lumps of clay and any objectionable foreign matter. **Sand.**

2. (*a*) The sand shall be well graded from coarse to fine and when tested for gradation as to size shall meet the following requirements: **Grading.**

Total passing ¼-in. screen	100 per cent.
Total passing a No. 10 sieve.....not less than	90 "

(*b*) These tests shall be made in accordance with the Standard Method of Mechanical Analysis of Sand or Other Fine Highway Material, except Fine Aggregates Used in Cement Concrete (Serial Designation: D 7) of the American Society for Testing Materials,[2] with the exception that a ¼-in. screen conforming to the requirements given in the Standard Method of Mechanical Analysis of Mixtures of Sand or Other Fine Material with Broken Stone or Broken Slag, Except Aggregates Used in Cement Concrete (Serial Designation: D 19) of the American Society for Testing Materials[3] shall be used in addition to the others specified in Method D 7.

3. (*a*) The sand shall be passed through a No. 10 sieve and that portion retained by the No. 10 sieve shall be dis- **Mortar Strength.**

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] See p. 479. [3] See p. 482.

carded. The portion passing a No. 10 sieve shall be mixed with portland cement conforming to the requirements of the Standard Specifications and Tests for Portland Cement (Serial Designation: C 9) of the American Society for Testing Materials,[1] in the proportion of 1 part cement to 3 parts of sand, by weight, according to the standard method of making 1:3 mortar briquets. The resulting mortar at the age of 7 and 28 days shall have a tensile strength of at least 75 per cent of that developed in the same time by mortar of the same consistency, made of 1 part of the same cement and 3 parts of Ottawa sand.

(*b*) Preliminary acceptance samples shall be subjected to both 7 and 28-day mortar tests, and acceptance based thereon. Samples tested during the progress of the work will be accepted on the basis of the 7-day test.

(*c*) The tests for mortar strength shall be made in accordance with the A.S.T.M. Standard Specifications and Tests for Portland Cement.

NOTE.—The portland cement used in testing the proposed sand should preferably be that used or to be used in the work.

Inspection. 4. The sand shall be subject to inspection after delivery at the place of use, prior to and during the laying of the pavement.

Sampling. 5. The sand shall be sampled for testing at the source of supply, if required. Samples shall also be taken for testing from time to time, at the place of use prior to and during the laying of the pavement, whenever desired by the engineer.

PORTLAND CEMENT FOR CEMENT MORTAR BED

Cement. 6. Portland cement for use in the cement mortar cushion shall conform to the requirements of the A.S.T.M. Standard Specifications and Tests for Portland Cement.[1]

[1] See p. 23.

STANDARD SPECIFICATIONS
FOR
BLOCK FOR GRANITE BLOCK PAVEMENTS[1]

Serial Designation: D 59 – 26

These specifications are issued under the fixed designation D 59; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1919; ADOPTED IN AMENDED FORM, 1922; REVISED, 1926.

General.

1. The blocks shall be of granite of medium-size grain, showing an even distribution of constituent minerals. They shall be of uniform quality and texture throughout, and free from seams or disintegrated materials.

PHYSICAL PROPERTIES AND TESTS

Physical Properties.

2. The average of three tests on sample blocks shall conform to the following requirements as to physical properties:

FOR HEAVY TRAFFIC.			
Percentage of wear	not more than		3.6
(French coefficient of wear	" less "		11)
Toughness	" less "		9
FOR MODERATELY HEAVY TRAFFIC.			
Percentage of wear	not more than		5.0
(French coefficient of wear	" less "		8)
Toughness	" less "		7

Methods of Testing.

3. (*a*) The percentage of wear and the French coefficient of wear shall be determined in accordance with the Standard Method of Test for Abrasion of Rock (Serial Designation: D 2) of the American Society for Testing Materials.[2]

(*b*) The toughness shall be determined in accordance with the Standard Method of Test for Toughness of Rock (Serial Designation: D 3) of the American Society for Testing Materials.[3]

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] See p. 463. [3] See p. 464.

DIMENSIONS

Dimensions.

4. The blocks shall conform to the following requirements as to dimensions:

Length on top, in	8 to 12
Width on top, in	$3\frac{1}{2}$ to $4\frac{1}{2}$ or $4\frac{1}{2}$ to $5\frac{1}{2}$
Depth, in	$4\frac{3}{4}$ to $5\frac{1}{4}$

DRESSING

Dressing.

5. (*a*) The blocks shall be so dressed that the faces will be approximately rectangular in shape, and the ends and sides sufficiently smooth to permit the blocks to be laid with joints not exceeding $\frac{1}{2}$ in. in width at the top, and for 1 in. downward therefrom, and not exceeding 1 in. in width at any other part of the joint.

(*b*) The wearing surface of the blocks shall show no depressions more than $\frac{3}{8}$ in. in depth, and the edges and corners shall be unchipped and unbroken.

INSPECTION AND REJECTION

Inspection.

6. At least six blocks shall be selected for physical tests by the engineer or his authorized representative, subsequent to delivery at the place of use, so as to fairly represent actual deliveries. No sample shall include blocks that would be rejected by a visual examination. The bedding plane shall be marked on at least two of the blocks selected.

Rejection.

7. All deliveries shall be subjected to further inspection at the place of use, prior to and during laying. All blocks which fail to conform to the requirements of Sections 1, 4 and 5 shall be rejected.

STANDARD SPECIFICATIONS
FOR
BLOCK FOR RECUT GRANITE BLOCK PAVEMENTS[1]

Serial Designation: D 131 – 23

These specifications are issued under the fixed designation D 131; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED IN AMENDED FORM, 1923.

General. 1. The blocks shall be of granite of medium-size grain, showing an even distribution of constituent minerals. They shall be of uniform quality and texture throughout, and free from seams or disintegrated materials.

PHYSICAL PROPERTIES AND TESTS

Physical Properties. 2. The average of three tests on sample blocks shall conform to the following requirements as to physical properties:

Percentage of wear	not more than 5
(French coefficient of wear	not less than 8)
Toughness	not less than 7

Methods of Testing. 3. (*a*) The percentage of wear and the French coefficient of wear shall be determined in accordance with the Standard Method of Test for Abrasion of Rock (Serial Designation: D 2) of the American Society for Testing Materials.[2]

(*b*) The toughness shall be determined in accordance with the Standard Method of Test for Toughness of Rock (Serial Designation: D 3) of the American Society for Testing Materials.[3]

DIMENSIONS

Dimensions. 4. The blocks shall conform to the following requirements as to dimensions:

Length on top, in.	**6 to 12**
Width on top, in.	**3½ to 4½**
Depth, in.	**4¾ to 5¼[a]**

[a] The depth may be varied by the engineer between 4 and 5 in., depth of blocks to suit the old material, provided that the blocks used on one contract shall not vary more than ¼ in. above or below the depth specified.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] See p. 463.] [3] See p. 464.

DRESSING

Dressing. 5. (*a*) The blocks shall be so dressed that the faces will be approximately rectangular in shape, and the ends and sides sufficiently smooth to permit the blocks to be laid with joints not exceeding $\frac{1}{2}$ in. in width at the top, and for 1 in. downward therefrom, and not exceeding 1 in. in width at any other part of the joint.

(*b*) The wearing surface of the blocks shall show no depressions more than $\frac{3}{8}$ in. in depth, and the edges and corners shall be unchipped and unbroken.

INSPECTION AND REJECTION

Inspection. 6. At least eight blocks shall be selected for physical tests by the engineer or his authorized representative, subsequent to delivery at the place of use, so as to fairly represent actual deliveries. No sample shall include blocks that would be rejected by a visual examination. The bedding plane shall be marked on at least two of the blocks selected.

Rejection. 7. All deliveries shall be subjected to further inspection at the place of use, prior to and during laying. All blocks which fail to conform to the requirements of Sections 1, 4 and 5 shall be rejected.

STANDARD SPECIFICATIONS
FOR
BLOCK FOR DURAX GRANITE PAVEMENTS[1]

Serial Designation: D 132 – 23

These specifications are issued under the fixed designation D 132; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED, 1923.

General.

1. The blocks shall be of granite of medium-size grain, showing an even distribution of constituent minerals. They shall be of uniform quality and texture throughout, and free from seams or disintegrated materials.

PHYSICAL PROPERTIES AND TESTS

Physical Properties.

2. The average of three tests on sample blocks shall conform to the following requirements as to physical properties:

Percentage of wear	not more than 5
(French coefficient of wear	not less than 8)
Toughness	not less than 7

Methods of Testing.

3. (*a*) The percentage of wear and the French coefficient of wear shall be determined in accordance with the Standard Method of Test for Abrasion of Rock (Serial Designation: D 2) of the American Society for Testing Materials.[2]

(*b*) The toughness shall be determined in accordance with the Standard Method of Test for Toughness of Rock (Serial Designation: D 3) of the American Society for Testing Materials.[3]

DIMENSIONS

Dimensions.

4. The blocks shall be not less than 3 in. nor more than 4 in. in length, width or depth.

DRESSING

Dressing.

5. (*a*) The blocks shall be so dressed that the faces will be approximately rectangular in shape, and the ends and sides sufficiently

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] See p. 463.

[3] See p. 464.

smooth to permit the blocks to be laid with joints not exceeding $\frac{1}{2}$ in. in width at the top, and for 1 in. downward therefrom, and not exceeding 1 in. in width at any other part of the joint.

(*b*) The wearing surface of the blocks shall show no depressions more than $\frac{3}{8}$ in. in depth, and the edges and corners shall be unchipped and unbroken.

INSPECTION AND REJECTION

Inspection. 6. At least 25 blocks shall be selected for physical tests by the engineer or his authorized representative, subsequent to delivery at the place of use, so as to fairly represent actual deliveries. No sample shall include blocks that would be rejected by a visual examination. The bedding plane shall be marked on at least two of the blocks selected.

Rejection. 7. All deliveries shall be subjected to further inspection at the place of use, prior to and during laying. All blocks which fail to conform to the requirements of Sections 1, 4 and 5 shall be rejected.

STANDARD SPECIFICATIONS

FOR

BROKEN SLAG FOR WATERBOUND BASE AND WEARING COURSE[1]

Serial Designation: D 65 – 23

These specifications are issued under the fixed designation D 65; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1920; ADOPTED, 1923.

Scope.

1. These specifications cover primarily slag screenings and coarse slag to be used in the construction of a waterbound base upon which some higher type of wearing course will be placed, preferably after the base has been firmly compacted by traffic. For a light traffic road it may also serve for both foundation and wearing course, provided the thickness of each is not less than 4 in.

General Characteristics.

2. The broken slag shall be air-cooled blast-furnace slag and shall consist of angular fragments reasonably uniform in density and quality, and reasonably free from thin, elongated or glassy pieces, dirt or other objectionable matter.

Weight.

3. The weight per cubic foot of each size specified, shall be not less than 65 lb.

Percentage of Wear.

4. The percentage of wear shall be not more than 12.0 per cent.

Screenings.

5. Screenings are that portion of the product of the crusher, including the dust of fracture, which, when tested by means of laboratory screens, shall meet the following requirements:

Passing ¾-in. screen	not less than 95 per cent
Passing ¼-in. screen	40 – 80 "

Coarse Slag.

6. Coarse slag is that portion of the product of the crusher which, when tested by means of laboratory screens, shall meet the following requirements:

Passing 4-in. screen	not less than 95 per cent
Retained on 2½-in. screen	" " " 85 "

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

Methods of Testing.

7. (*a*) *Weight per Cubic Foot.*—The weight per cubic foot shall be determined as specified in the Standard Method of Test for Unit Weight of Aggregate for Concrete (Serial Designation: C 29) of the American Society for Testing Materials.[1]

(*b*) *Percentage of Wear.*—The percentage of wear shall be determined as specified in the Standard Method of Test for Abrasion of Rock (Serial Designation: D 2) of the American Society for Testing Materials,[2] using, however, a volume of 4000 cc. measured loose of the broken slag of the size specified in the standard test.

Sampling.

8. The method of sampling shall conform to the Standard Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods (Serial Designation: D 75) of the American Society for Testing Materials.[3]

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[1] See p. 120.
[2] See p. 463.
[3] See p. 473.

STANDARD SPECIFICATIONS
FOR
SHOVEL-RUN OR CRUSHER-RUN BROKEN SLAG FOR WATERBOUND BASE[1]

Serial Designation: D 66 – 23

These specifications are issued under the fixed designation D 66; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1920; ADOPTED, 1923.

Scope.

1. These specifications cover primarily slag to be used in the construction of a waterbound base upon which some higher type of wearing course will be placed, preferably after the base has been compacted by traffic.

General Characteristics.

2. The broken slag shall be air-cooled blast-furnace slag and shall consist of angular fragments reasonably uniform in density and quality, and reasonably free from thin, elongated or glassy pieces, dirt or other objectionable matter.

Weight.

3. The weight per cubic foot shall be not less than 65 lb.

Percentage of Wear.

4. The percentage of wear shall be not more than 15.0 per cent.

Grading.

5. Shovel-run or crusher-run slag, when tested by means of laboratory screens, shall meet the following requirements:

Passing 4-in. screen	not less than 95 per cent
Passing 2½-in. screen	25 – 75 "

Methods of Testing.

6. (*a*) *Weight per Cubic Foot.*—The weight per cubic foot shall be determined as specified in the Standard Method of Test for Unit Weight of Aggregate for Concrete (Serial Designation: C 29) of the American Society for Testing Materials.[2]

(*b*) *Percentage of Wear.*—The percentage of wear shall be determined as specified in the Standard Method of Test for Abrasion of Rock (Serial Designation: D 2) of the American Society for Testing Materials,[3] using, however, a volume of 4000 cc. measured loose of the broken slag of the size specified in the standard test.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] See p. 120.

[3] See p. 463.

Sampling.

7. The method of sampling shall conform to the Standard Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods (Serial Designation: D 75) of the American Society for Testing Materials.[1]

[1] See p. 473.

STANDARD SPECIFICATIONS

FOR

BROKEN SLAG FOR BITUMINOUS MACADAM BASE[1]

Serial Designation: D 195 – 27

These specifications are issued under the fixed designation D 195; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1924; ADOPTED, 1927.

Scope.

1. These specifications cover $\frac{3}{4}$ to $1\frac{1}{4}$-in., $1\frac{1}{4}$ to $2\frac{1}{2}$-in., and $2\frac{1}{2}$ to $3\frac{1}{2}$-in. size broken slag to be used in the construction of a bituminous macadam base in which the bituminous material is applied by the penetration method and which is to be covered with any type of surface course.

The $2\frac{1}{2}$ to $3\frac{1}{2}$-in. size is intended for use as a first course, which will be penetrated with bituminous material, when a base of over $3\frac{1}{2}$ in. is desired.

The $1\frac{1}{4}$ to $2\frac{1}{2}$-in. size is intended for use as a second course which is to be penetrated with bituminous material.

The $\frac{3}{4}$ to $1\frac{1}{4}$-in. size is intended to fill the surface voids when a sheet asphalt or a fine-graded bituminous concrete is to be placed directly upon it. This material can be omitted when a coarse-graded bituminous concrete or a binder course is to be laid directly upon the base.

General Characteristics.

2. The broken slag shall be air-cooled blast-furnace slag and shall consist of angular fragments reasonably uniform in density and quality, and reasonably free from thin, elongated or glassy pieces, dirt or other objectionable matter.

Weight.

3. The weight per cubic foot of each size specified shall not be less than 65 lb.

$2\frac{1}{2}$ to $3\frac{1}{2}$-in. and $1\frac{1}{4}$ to $2\frac{1}{2}$-in. Size.

4. The coarse slag when tested by means of laboratory screens shall conform to the following requirements:

(*a*) Passing $3\frac{1}{2}$-in. screen	not less than 95 per cent
Total passing $2\frac{1}{2}$-in. screen	not more than 15 "
(*b*) Passing $2\frac{1}{2}$-in. screen	not less than 95 per cent
Total passing $1\frac{1}{4}$-in. screen	not more than 15 "

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

¾ to 1¼-in. Size.

5. The intermediate size slag when tested by means of laboratory screens shall conform to the following requirements:

Passing 1¼-in. screen	not less than 95 per cent
Passing 1-in. screen	25 to 75 "
Passing ¾-in. screen	not more than 15 "

Methods of Testing.

6. *Weight per Cubic Foot.*—The weight per cubic foot shall be determined in accordance with the Standard Method of Test for Unit Weight of Aggregate for Concrete (Serial Designation: C 29) of the American Society for Testing Materials.[1]

Sampling.

7. The method of sampling shall conform to the Standard Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods (Serial Designation: D 75) of the American Society for Testing Materials.[2]

NOTE.—The test values for quality of slag incorporated in these specifications are suggested as the lowest safe requirements. It is not expected, however, that the limits for size and quality will be applicable to all conditions.

[1] See p. 120.

[2] See p. 473.

STANDARD SPECIFICATIONS
FOR
BROKEN SLAG FOR BITUMINOUS CONCRETE BASE[1]

Serial Designation: D 196 – 27

These specifications are issued under the fixed designation D 196; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1924; ADOPTED, 1927.

Scope.

1. These specifications cover $\frac{1}{4}$ to $1\frac{1}{4}$-in. and $\frac{1}{4}$ to $2\frac{1}{2}$-in. size broken slag, either of which is to be used in the construction of a bituminous concrete base prepared by the mixing method and upon which may be placed any type of surface course.

General Characteristics.

2. The broken slag shall be air-cooled blast-furnace slag and shall consist of angular fragments reasonably uniform in density and quality, and reasonably free from thin, elongated or glassy pieces, dirt or other objectionable matter.

Weight.

3. The weight per cubic foot of each size specified shall not be less than 70 lb.

$\frac{1}{4}$ to $1\frac{1}{4}$-in. or $\frac{1}{4}$ to $2\frac{1}{2}$-in. Size.

4. The slag when tested by means of laboratory screens shall conform to one of the following requirements:

(*a*)	Passing $1\frac{1}{4}$-in. screen	not less than 95 per cent
	Passing $\frac{3}{4}$-in. screen	25 to 75 "
	Passing $\frac{1}{4}$-in. screen	not more than 5 "
(*b*)	Passing $2\frac{1}{2}$-in. screen	not less than 95 per cent
	Passing $1\frac{1}{4}$-in. screen	25 to 75 "
	Passing $\frac{1}{4}$-in. screen	not more than 5 "

NOTE.—One of the above alternate sizes for broken slag should be specified to best suit the local and service conditions.

Methods of Testing.

5. *Weight per Cubic Foot.*—The weight per cubic foot shall be determined in accordance with the Standard Method of Test for Unit Weight of Aggregate for Concrete (Serial Designation: C 29) of the American Society for Testing Materials.[2]

Sampling.

6. The methods of sampling shall conform to the Standard Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods (Serial Designation: D 75) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] See p. 120.

[3] See p. 473.

STANDARD SPECIFICATIONS

FOR

BROKEN SLAG FOR BITUMINOUS MACADAM WEARING COURSE[1]

Serial Designation: D 159 - 27

These specifications are issued under the fixed designation D 159; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED IN AMENDED FORM, 1927.

Scope.

1. These specifications cover $\frac{3}{4}$ to $\frac{1}{4}$-in., $1\frac{1}{4}$ to $\frac{3}{4}$-in., and $2\frac{1}{2}$ to $1\frac{1}{4}$-in. or $3\frac{1}{2}$ to $2\frac{1}{2}$-in. size slag to be used in the construction of a bituminous macadam wearing course in which the bituminous material is applied by the penetration method. The maximum size of $3\frac{1}{2}$ in. specified for the wearing course is on the basis of a course $2\frac{1}{2}$ to $3\frac{1}{2}$ in. in thickness after consolidation. The $1\frac{1}{4}$ to $\frac{3}{4}$-in. size should be used to fill the surface voids after the first application of bituminous material, and the $\frac{3}{4}$ to $\frac{1}{4}$-in. size after the application of the seal coat.

General Characteristics.

2. The broken slag shall be air-cooled blast-furnace slag and shall consist of angular fragments reasonably uniform in density and quality, and reasonably free from thin, elongated or glassy pieces, dirt or other objectionable matter.

Weight.

3. The weight per cubic foot of each size specified shall not be less than 70 lb.

$\frac{3}{4}$ to $\frac{1}{4}$-in. Size.

4. The $\frac{3}{4}$ to $\frac{1}{4}$-in. size is that portion of the product of the crusher, which, when tested by means of laboratory screens, shall meet the following requirements:

Passing $\frac{3}{4}$-in. screen	not less than 95 per cent
Passing $\frac{1}{2}$-in. screen	25 to 75 "
Passing $\frac{1}{4}$-in. screen	not more than 15 "

$1\frac{1}{4}$ to $\frac{3}{4}$-in. Size.

5. The $1\frac{1}{4}$ to $\frac{3}{4}$-in. size is that portion of the product of the crusher, which, when tested by means of laboratory screens, shall meet the following requirements.

Passing $1\frac{1}{4}$-in. screen	not less than 95 per cent
Passing 1-in. screen	25 to 75 "
Passing $\frac{3}{4}$-in. screen	not more than 15 "

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

6. The $2\frac{1}{2}$ to $1\frac{1}{4}$-in. and $3\frac{1}{2}$ to $2\frac{1}{2}$-in. sizes are that portion of the product of the crusher, which, when tested by means of laboratory screens, shall conform to one of the following requirements: $2\frac{1}{2}$ to $1\frac{1}{4}$-in. and $3\frac{1}{2}$ to $2\frac{1}{2}$-in. Size.

Passing $2\frac{1}{2}$-in. screen	not less than 95 per cent
Total passing $1\frac{1}{4}$-in. screen	not more than 15 "
Passing $3\frac{1}{2}$-in. screen	not less than 95 per cent
Total passing $2\frac{1}{2}$-in. screen	not more than 15 "

NOTE.—One of the above alternate sizes for coarse slag should be specified to best suit the local and service conditions.

7. *Weight per Cubic Foot.*—The weight per cubic foot shall be determined in accordance with the Standard Method of Test for Unit Weight of Aggregate for Concrete (Serial Designation: C 29) of the American Society for Testing Materials.[1] Methods of Testing.

8. The method of sampling shall conform to the Standard Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods (Serial Designation: D 75) of the American Society for Testing Materials.[2] Sampling.

[1] See p. 120.
[2] See p. 473.

STANDARD SPECIFICATIONS
FOR
BROKEN SLAG FOR BITUMINOUS CONCRETE (COARSE-GRADED AGGREGATE TYPE)[1]

Serial Designation: D 160 - 27

These specifications are issued under the fixed designation D 160; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED IN AMENDED FORM, 1927.

Scope.

1. These specifications cover $\frac{3}{4}$ to $\frac{1}{4}$-in. and $1\frac{1}{4}$ to $\frac{1}{4}$-in. size slag to be used in the construction of a coarse-graded bituminous concrete wearing course constructed by the mixing method. The $\frac{3}{4}$ to $\frac{1}{4}$-in. size should be used after the application of the seal coat when a seal coat is used.

General Characteristics.

2. The broken slag shall be air-cooled blast-furnace slag and shall consist of angular fragments reasonably uniform in density and quality, and reasonably free from thin, elongated or glassy pieces, dirt or other objectionable matter.

Weight.

3. The weight per cubic foot of each size specified shall not be less than 70 lb.

$\frac{3}{4}$ to $\frac{1}{4}$-in. Size.

4. The $\frac{3}{4}$ to $\frac{1}{4}$-in. size is that portion of the product of the crusher, which, when tested by means of laboratory screens, shall meet the following requirements:

Passing $\frac{3}{4}$-in. screen	not less than 95 per cent
Passing $\frac{1}{2}$-in. screen	25 to 75 "
Passing $\frac{1}{4}$-in. screen	not more than 15 "

$1\frac{1}{4}$ to $\frac{1}{4}$-in. Size.

5. The $1\frac{1}{4}$ to $\frac{1}{4}$-in. size is that portion of the product of the crusher, which, when tested by means of laboratory screens, shall meet the following requirements:

Passing $1\frac{1}{4}$-in. screen	95 to 100 per cent
Passing $\frac{3}{4}$-in. screen	25 to 75 "
Passing $\frac{1}{4}$-in. screen	not more than 5 "

Methods of Testing.

6. *Weight per Cubic Foot.*—The weight per cubic foot shall be determined in accordance with the Standard Method of Test for

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

Unit Weight of Aggregate for Concrete (Serial Designation: C 29) of the American Society for Testing Materials.[1]

Sampling. 7. The method of sampling shall conform to the Standard Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods (Serial Designation: D 75) of the American Society for Testing Materials.[2]

[1] See p. 120.
[2] See p. 473.

STANDARD SPECIFICATIONS

FOR

BROKEN SLAG FOR BITUMINOUS CONCRETE (FINE-GRADED AGGREGATE TYPE)[1]

Serial Designation: D 161 - 27

These specifications are issued under the fixed designation D 161; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED IN AMENDED FORM, 1927.

Scope.

1. These specifications cover slag aggregate to be used in the construction of a bituminous concrete wearing surface constructed by the mixing method.

General Characteristics.

2. The broken slag shall be air-cooled blast-furnace slag and shall consist of angular fragments reasonably uniform in density and quality, and reasonably free from thin, elongated or glassy pieces, dirt or other objectionable matter.

Weight.

3. The weight per cubic foot shall be not less than 70 lb.

Slag Aggregate 0 to ½-in Size.

4. The slag aggregate is that portion of the product of the crusher, which, when tested by means of laboratory screens, shall conform to the following requirements:

Passing ½-in. screen	95 to 100 per cent
Passing ¼-in. screen	0 to 75 "

Methods of Testing.

5. *Weight per Cubic Foot.*—The weight per cubic foot shall be determined in accordance with the Standard Method of Test for Unit Weight of Aggregate for Concrete (Serial Designation: C 29) of the American Society for Testing Materials.[2]

Sampling.

6. The method of sampling shall conform to the Standard Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods (Serial Designation: D 75) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] See p. 120.

[3] See p. 473.

STANDARD METHOD OF TEST
FOR
ABRASION OF ROCK[1]

Serial Designation: D 2 – 26

This method is issued under the fixed designation D 2; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1908; REVISED, 1926.

This well-known method of test is similar in almost all respects to the Deval abrasion test of the French School of Roads and Bridges. It has been used since 1878, and is entirely satisfactory for the purpose for which it was designed.

Apparatus. 1. The machine shall consist of one or more hollow iron cylinders; closed at one end and furnished with a tightly fitting iron cover at the other; the cylinders to be 20 cm. in diameter and 34 cm. in depth, inside. These cylinders are to be mounted on a shaft at an angle of 30 deg. with the axis of rotation of the shaft.

Preparation of Sample. 2. At least 30 lb. of coarsely broken stone shall be available for a test. The rock to be tested shall be broken in pieces as nearly uniform in size as possible, and as nearly 50 pieces as possible shall constitute a test sample.

Procedure. 3. The total weight of rock in a test shall be within 10 g. of 5 kg. All test pieces shall be washed and thoroughly dried before weighing. Ten thousand revolutions, at a rate of between 30 and 33 per minute, shall constitute a test. Only the percentage of material worn off which will pass through a 0.16-cm. ($\frac{1}{16}$-in.) mesh sieve shall be considered in determining the amount of wear. This may be expressed either as the percentage of the 5 kg. used in the test, or the French coefficient, which is in more general use, may be given; that is, coefficient of wear $= 20 \times \frac{20}{w} = \frac{400}{w}$ where w is the weight in grams of the detritus under 0.16 cm. ($\frac{1}{16}$ in.) in size per kilogram of rock used.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

STANDARD METHOD OF TEST
FOR
TOUGHNESS OF ROCK[1]

Serial Designation: D 3 – 18

This method is issued under the fixed designation D 3; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1908; REVISED, 1918.

This method was approved March 28, 1921, as "Tentative American Standard" by the American Engineering Sandards Committee.

Definition.

1. Toughness, as applied to rock, is the resistance offered to fracture under impact, expressed as the final height of blow required of a standard hammer to cause fracture of a cylindrical test specimen of given dimensions.

Impact Machine.

2. Any form of impact machine which will comply with the following essentials may be used in making the test:

(*a*) A cast-iron anvil weighing not less than 50 kg., firmly fixed upon a solid foundation;

(*b*) A hammer weighing 2 kg., arranged so as to fall freely between suitable guides;

(*c*) A plunger made of hardened steel and weighing 1 kg., arranged to slide freely in a vertical direction in a sleeve, the lower end of the plunger being spherical in shape with a radius of 1 cm.;

(*d*) Means for raising the hammer and for dropping it upon the plunger from any specified height from 1 to not less than 75 cm., and means for determining the height of fall to approximately 1 mm.;

(*e*) Means for holding the cylindrical test specimen securely on the anvil without rigid lateral support, and under the plunger in such a way that the center of its upper surface shall, throughout the test, be tangent to the spherical end of the plunger at its lowest point.

Sampling.

3. Quarry samples of rock from which test specimens are to be prepared shall measure at least 6 in. on a side and at least 4 in. in thickness, and when possible shall have the plane of structural weakness of the rock plainly marked thereon.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction, of the A.S.T.M. Committee D-4 on Road and Paving Materials.

Samples should be taken from freshly quarried material, and only from pieces which show no evidences of incipient fracture due to blasting or other causes. The samples should preferably be split from large pieces by the use of plugs and feathers and not by sledging. Commercial stone-block samples from which test specimens are to be prepared shall measure at least 3 in. on each edge.

Size and Form of Test Specimens.

4. Specimens for test shall be cylinders prepared as described in Section 5, 25 mm. in height and from 24 to 25 mm. in diameter. Three test specimens shall constitute a test set. The ends of the specimen shall be plane surfaces at right angles to the axis of the cylinder.

Preparation of Test Specimens.

5. One set of specimens shall be drilled perpendicular and another parallel to the plane of structural weakness of the rock, if such plane is apparent. If a plane of structural weakness is not apparent, one set of specimens shall be drilled at random. Specimens shall be drilled in a manner which will not subject the material to undue stresses and which will insure the specified dimensions.[1] The ends of the cylinders may be sawed by means of a band or diamond saw,[2] or in any other way which will not induce incipient fracture, but shall not be chipped or broken off with a hammer. After sawing, the ends of the specimens shall be ground plane with water and carborundum or emery on a cast-iron lap until the cylinders are 25 mm. in length.

Method of Testing.

6. The test shall consist of a 1-cm. fall of the hammer for the first blow, a 2-cm. fall for the second blow, and an increase of 1-cm. fall for each succeeding blow until failure of the test specimen occurs.

Recording and Reporting Results.

7. The height of the blow in centimeter at failures shall be the toughness of the test specimen. The individual and the average toughness of three test specimens shall be reported when no plane of structural weakness is apparent. In cases where a plane of structural weakness is apparent, the individual and average toughness of the three specimens in each set shall be reported and identified. Any peculiar condition of a test specimen which might affect the result, such as the presence of seams, fissures, etc., shall be noted and recorded with the test result.

[1] The form of diamond drill described in *Bulletin No. 347*, U. S. Department of Agriculture, pp. 6–7, is recommended, and should prove satisfactory if the instructions are strictly followed.

[2] A satisfactory form of diamond saw is described in *Bulletin No. 347*, U. S. Department of Agriculture, pp. 7–9.

STANDARD METHOD OF TEST FOR APPARENT SPECIFIC GRAVITY OF COARSE AGGREGATES[1]

Serial Designation: D 30 – 18

This method is issued under the fixed designation D 30; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1916; ADOPTED, 1918.

This method was approved July 18, 1924, as "Tentative American Standard" by the American Engineering Standards Committee.

The apparent specific gravity shall be determined in the following manner:

1. The sample, weighing 1000 g. and composed of pieces approximately cubical or spherical in shape and retained on a screen having 1.27-cm. ($\frac{1}{2}$-in.) circular openings, shall be dried to constant weight at a temperature between 100 and 110° C. (212 and 230° F.), cooled, and weighed to the nearest 0.5 g. Record this weight as weight *A*. In the case of homogeneous material, the smallest particles in the sample may be retained on a screen having $1\frac{1}{4}$-in. circular openings.

2. Immerse the sample in water for 24 hours, surface-dry individual pieces with aid of a towel or blotting paper, and weigh. Record this weight as weight *B*.

3. Place the sample in a wire basket of approximately $\frac{1}{4}$-in. mesh, and about 12.7 cm. (5 in.) square and 10.3 cm. (4 in.) deep, suspend in water[2] from center of scale pan, and weigh. Record the difference between this weight and the weight of the empty basket suspended in water as weight *C*. (Weight of saturated sample immersed in water.)

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] The basket may be conveniently suspended by means of a fine wire hung from a hook shaped in the form of a question mark with the top end resting on the center of the scale pan.

4. The apparent specific gravity shall be calculated by dividing the weight of the dry sample (*A*) by the difference between the weights of the saturated sample in air (*B*) and in water (*C*), as follows:

$$\text{Apparent Specific Gravity} = \frac{A}{B - C}.$$

5. Attention is called to the distinction between apparent specific gravity and true specific gravity. Apparent specific gravity includes the voids in the specimen and is therefore always less than or equal to, but never greater than the true specific gravity of the material.

STANDARD METHODS OF TEST
FOR
APPARENT SPECIFIC GRAVITY OF SAND, STONE AND SLAG SCREENINGS, AND OTHER FINE NON-BITUMINOUS HIGHWAY MATERIALS[1]

Serial Designation: D 55 – 25

These methods are issued under the fixed designation D 55; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1918; ADOPTED, 1919; REVISED, 1924, 1925.

Alternate Methods.

1. The following tests, "Le Chatelier" and "Jackson," are equally suited for use in determining the apparent specific gravity of sand, stone and slag screenings and other fine non-bituminous highway materials, and may be considered as alternates:

LE CHATELIER TEST.

Apparatus.

2. The determination of specific gravity shall be made with a standardized Le Chatelier apparatus which conforms to the requirements illustrated in Fig. 1. This apparatus is standardized by the United States Bureau of Standards. Kerosine or water may be used in making this determination.

Method.

3. (*a*) The flask shall be filled with either of these liquids to a point on the stem between zero and one cubic centimeter and a reading taken. From 50 to 64 g. (depending upon its approximate specific gravity and to bring the reading on the scale) of sand or other fine non-bituminous highway material of the same temperature as the liquid, shall be slowly introduced, taking care that the material does not adhere to the inside of the flask above the liquid and to free the material from air by rolling the flask in an inclined position. After all material is introduced, the level of the liquid in the graduated stem shall be read and the difference between readings is the volume displaced by the known weight of the material.

Calculation.

(*b*) The specific gravity shall then be calculated from the formula:

$$\text{Specific gravity} = \frac{\text{Weight of material (g.)}}{\text{Displaced volume (cc.)}}$$

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

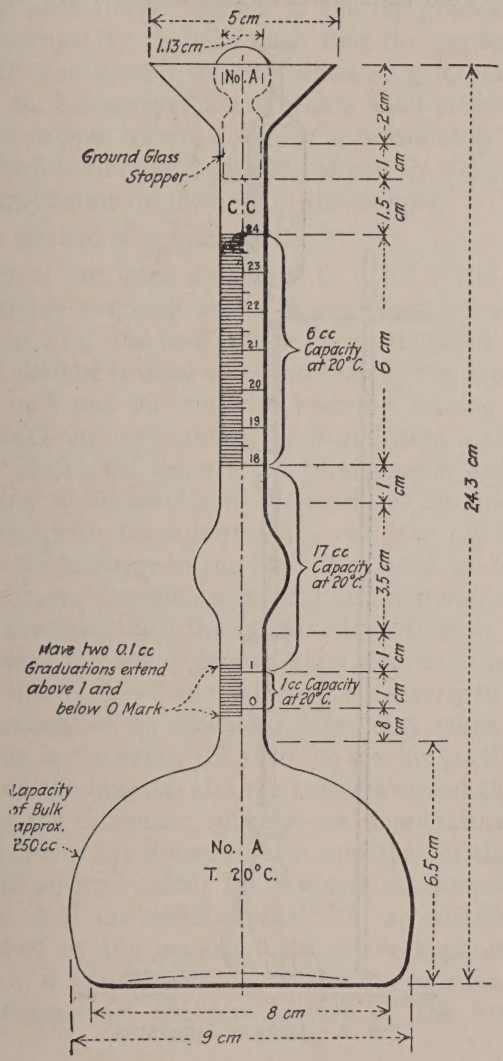


FIG. 1.—Le Chatelier Apparatus.

(*c*) The flask, during the operation, shall be kept immersed in water, in order to avoid variations in the temperature of the liquid in the flask, which shall not exceed 0.5° C. The results of repeated tests should agree within 0.01.

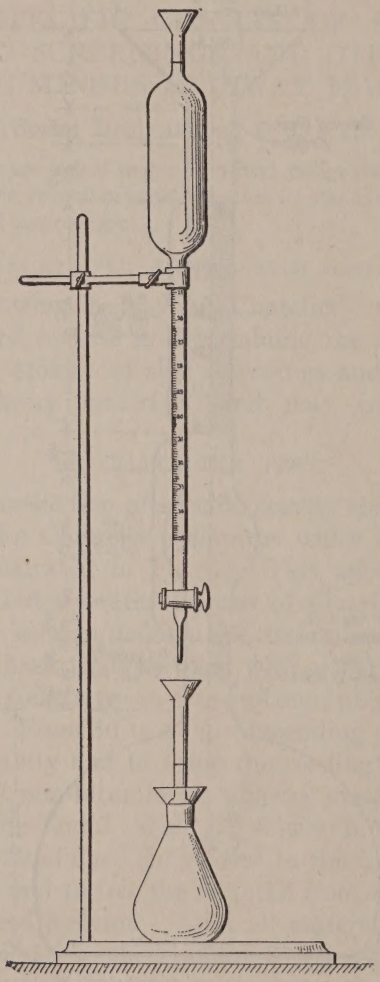


FIG. 2.—Jackson Apparatus.

JACKSON TEST.

Apparatus. 4. The determination shall be made with the Jackson specific gravity apparatus (illustrated in Fig. 2) which shall consist of a burette, with graduations reading to 0.02 in specific gravity, about 28 cm. (11 in.) in length and with an inside diameter of

about 0.7 cm. (0.3 in.), which shall be connected with a glass bulb approximately 13 cm. (5⅛ in.) in length and 4.5 cm. (1.75 in.) in diameter, the glass bulb being of such size that from a mark on the neck above the bulb to the top graduation on the burette, the capacity shall be such that the graduations shall read directly the specific gravity when 50 g. of materials are used; and an Erlenmeyer flask, which shall contain a hollow ground-glass stopper having a neck of approximately 6 to 8-mm. bore and which shall have a capacity of exactly 200 cc. (6.76 oz.) up to the graduation on the neck of the stopper.

5. The method is as follows: **Method.**

(1) Dry at not more than 110° C. (230° F.) to a constant weight a sample weighing about 55 g.; (2) weigh 50 g. of the dry sample to 0.1 g. and pour it into the unstoppered Erlenmeyer flask, which shall be cleaned and dried before each determination; (3) fill the bulb and burette with kerosine, leaving just space enough to take the temperature by introducing a thermometer through the neck; (4) remove the thermometer and add sufficient kerosine to fill exactly to the mark on the neck, drawing off any excess with the burette; (5) run into the flask about one-half of the kerosine in the bulb to remove air bubbles and then run in more kerosine, removing any material adhering to the neck of the flask, until the kerosine is just below the ground glass; (6) place the hollow ground-glass stopper in position and turn it to fit tightly, and then run in kerosine exactly to the 200-cc. (6.76-oz.) graduation on the neck, care being taken to remove all air bubbles in the flask; (7) read the specific gravity from the graduation on the burette, and the temperature of the oil in the flask, noting the difference between the temperature of the oil in the bulb before the determination and that of the oil in the flask after the determination; (8) make a temperature correction to the reading of the specific gravity in accordance with the table furnished by the manufacturer of the apparatus, adding the correction if the temperature of the kerosine has increased and subtracting it if the temperature of the kerosine has decreased.

STANDARD METHOD OF TEST FOR QUANTITY OF CLAY AND SILT IN GRAVEL FOR HIGHWAY CONSTRUCTION[1]

Serial Designation: D 72 – 21

This method is issued under the fixed designation D 72; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1920; ADOPTED, 1921.

Scope.

1. This test covers the determination of the quantity of clay and silt in natural gravel to be used in highway construction.

Treatment of Sample.

2. The sample as received shall be moistened and thoroughly mixed, then dried to constant weight at a temperature between 100 and 110° C. (212 and 230° F.).

Apparatus.

3. The pan or vessel to be used in the determination shall be 12 in. (30.5 cm.) in diameter by not less than 4 in. (10.2 cm.) deep, as nearly as may be obtained.

Procedure.

4. (*a*) A representative portion of the dry material weighing not less than 50 times the weight of the largest stone in the sample shall be selected from the sample and placed in the pan which has been dried and accurately weighed.

(*b*) Sufficient water shall be poured into the pan to cover the gravel. The gravel shall be agitated vigorously for 15 seconds with a trowel or stirring rod. After it has settled for 15 seconds the water shall be poured off into a tared evaporating dish, care being taken not to pour off any gravel. This is repeated until the wash water is clear. The washed material shall be dried to constant weight in an oven at a temperature between 100 and 110° C. (212 and 230° F.), weighed, and the net weight of gravel determined.

Percentage of Clay.

5. The percentage of clay and silt shall be calculated from the formula:

$$\text{Percentage of Clay and Silt} = \frac{\text{Original weight} - \text{weight after washing}}{\text{Original weight}} \times 100$$

Check Determination.

6. For a check on the results, the wash water shall be evaporated to dryness and the residue weighed:

$$\text{Percentage of Clay and Silt} = \frac{\text{Weight of residue}}{\text{Original weight}} \times 100.$$

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

STANDARD METHODS

OF

SAMPLING STONE, SLAG, GRAVEL, SAND AND STONE BLOCK FOR USE AS HIGHWAY MATERIALS,

INCLUDING SOME MATERIAL SURVEY METHODS[1]

Serial Designation: D 75 – 22

These methods are issued under the fixed designation D 75; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1920; ADOPTED, 1922.

These methods were approved July 18, 1924, as "Tentative American Standard" by the American Engineering Standards Committee.

1. Samples of all materials for test upon which is to be based the acceptance or rejection of the supply shall be taken by the engineer or his authorized representative. Samples for inspection or preliminary test may be submitted by a producer or owner of the supply.

SAMPLING OF STONE

A. Sampling of Stone from Ledges or Quarries for Quality.

Inspection.

2. The ledge or quarry face shall be inspected closely to determine any variation in different layers. Any difference in color or structure shall be observed, and if necessary to secure unweathered specimens, pieces broken from different layers.

Sampling and Size.

3. (*a*) For standard stone test, separate samples shall be taken of at least 30 lb. each of fresh unweathered specimens from all layers that appear to vary in color or structure. When more than one piece is taken, the minimum size shall be 2 in., except that there shall be one piece of each sample of a minimum size of 4 by 5 by 3 in. on which the bedding plane is marked and which shall be free from seams or fractures to be used in the toughness or compression test.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

(*b*) The sample for concrete test will depend on the kind of tests to be made and the number of specimens necessary.

Record. 4. In addition to the general information accompanying all samples, samples from local ledges not commercial sources shall contain the following: Name of owner; approximate quantity available (if quantity is very large this can be recorded as practically unlimited); amount and character of overburden or stripping; haul to nearest point on road where the material is to be used; character of haul (kind of road and grade); also some detailed record of the extent and location of the material represented by each sample. For this purpose a sketch, plan and elevation showing the thickness and location of the different layers is recommended.

B. Sampling of Stone from Commercial Quarries.

For Quality. 5. Where practical, samples from commercial quarries shall be taken from the ledge or quarry face and the same procedure shall be followed as when sampling local ledges.

For Size. 6. At the plant, the following factors affect the size of broken stone: Size and shape of screen openings; length of screen sections; the angle of screen with horizontal; the speed of screen rotation; and the rate at which the screen is fed. A general inspection and record of these conditions shall be made. The sample shall be taken preferably from cars or boats while loading from stock piles or bins. It is recommended, in order that the sample may be representative, that separate samples be taken at different times while the material is being loaded. If the sample has to be taken from the bin or stock pile, several samples shall be taken from different parts of the stock pile and from the top of the bin and the loading chute. These separate samples shall be well mixed in a composite sample and the sample for test obtained by the quartering method.

Sampling at Delivery. 7. (*a*) Where it is not practical to visit the plant, samples for both quality and size shall be taken from different parts of the car or boat during unloading. It is recommended that separate samples be taken from the top, middle and bottom of car or boat. These separate samples shall be well mixed in a composite sample and the sample for test obtained by the quartering method. The results of tests on crushed stone for quality are not considered comparable with results from samples specially broken for test in the laboratory, but the general quality of the stone can be ascertained and check tests on

various shipments should indicate any change in quality. In this case, tests for both quality and size can be made on the same sample.

(*b*) Where test is to be made for size only, it is recommended that a small set of screens and a pair of scales or some unit measure receptacle for measuring volume be used for field testing in order not to delay decision on the use of the material. Occasional check tests can be obtained from the laboratory to assure a fair degree of accuracy in field testing.

Size of Samples.

8. (*a*) The sample of crushed stone for mechanical analysis shall weigh at least 50 times the weight of the largest piece therein.

(*b*) The sample for concrete test will depend on the kind of tests to be made and the number of specimens necessary.

C. Sampling of Field Stone and Boulders.

Inspection.

9. A detailed inspection of the deposits of field stone and boulders, over the area where the supply is to be obtained, shall be made. The different kinds of stone and its state of preservation in the various deposits shall be recorded.

Separate Samples.

10. Separate samples shall be taken of all stone of different classes that a visual inspection indicates, from state of preservation and degrees of lamination, would be considered for use in construction.

Record.

11. Records accompanying samples of field stone and boulders, in addition to general information, shall contain the following:

(*a*) Location of supply;

The plotting of the field stone and boulder area on a U. S. topographic or a similar map is recommended for this purpose.

(*b*) Approximate quantity available;

A fairly accurate estimate of amount of stone in fences can be made by measuring a cross-section and pacing the length. It is more difficult to estimate the amount of stone scattered on the ground and ordinarily such deposits do not pay to work unless quite congested.

(*c*) Information regarding the percentage of different classes of stone which were sampled and the percentages of material which can be rejected by visual examination and may have to be handled and spoiled.

This information regarding the percentages of different kinds of material can only be estimated and the degree of accuracy attained will depend almost entirely upon the experience and skill of the individual.

SAMPLING OF BLAST-FURNACE SLAG

For Size and Quality.

12. It is recommended that blast-furnace slag be sampled for size and quality by the method specified for broken stone.

SAMPLING OF SAND AND GRAVEL

A. Non-Commercial Deposits.

Definition.

13. Non-commercial as used here includes all undeveloped sand and gravel deposits and all developed deposits where the material is not washed or screened.

Sampling.

14. (*a*) The investigator should realize that few if any natural sand and gravel deposits are uniform, and when a sample is taken the quantity of material in the deposit similar to the sample shall be ascertained as nearly as possible.

(*b*) Where possible, samples of sand shall be taken when it is in a damp condition.

(*c*) If the deposit is worked as a bank or pit and has an open face, the sample shall be taken by channeling the open face so as to represent material that visual inspection indicates could be used. Care shall be taken to eliminate any overburden or stripping at the top or any that has fallen along the face from the top. It is necessary, especially in small deposits, to excavate test pits some distance back of and parallel to the face to determine the extent of the supply. The number and depth of these pits depend on the quantity of material that is to be taken from the deposit. Separate samples shall be taken from the face of the bank and from test pits. These shall be well mixed in a composite sample and the sample for test obtained by the quartering method.

(*d*) Deposits that have no open face shall be sampled by means of test pits. The number and depth of these will depend on local conditions and the amount of material to be used from the source. A separate sample shall be taken from each pit and where visual examination indicates no radical difference in size of grain, color, etc., these shall be well mixed in a composite sample and the sample for test obtained by the quartering method. Where visual inspection indicates a distinct difference in material from different pits, separate samples shall be taken for test.

(*e*) It is suggested that the colorimetric test be used for determining the percentage of organic material.[1]

[1] For a description of this test, see the Standard Method of Test for Organic Impurities in Sands for Concrete (Serial Designation: C 40) of the American Society for Testing Materials, p. 123.

15. In addition to the general information accompanying all samples from sand and gravel deposits not commercial sources, the same detailed information shall be supplied as with samples of stone taken from local ledges. **Record.**

B. Commercial Sand and Gravel Plants.

16. Samples of both sand and gravel from commercial screening or washing plants shall be taken from the bins or storage piles preferably while the material is being loaded from these supplies. The sand and gravel shall be sampled as separate units and shipped as such. It is difficult to secure a representative sample from stock piles and bins and, if conditions require sampling from these sources, the following is recommended: Separate samples shall be taken from different parts of the stock pile, care being taken to avoid sampling a segregated area of coarse-grained material which is likely to exist at the base of the pile. In sampling from a bin, separate samples shall be taken from the top and the loading chute. At the latter place at least ½ cu. yd. of material shall be run off and representative samples taken from it. These separate samples shall be well mixed in a composite sample and the sample for test obtained by the quartering method. **Sampling for Quality.**

17. The size of sand and gravel will be affected by the same factors as broken stone and the same inspection is necessary. In addition, the relative amount of water used in screening must be taken into consideration. The same procedure for taking a sample of broken stone for size is recommended for sampling sand and gravel for size, both at plant and at delivery. **Sampling for Size.**

18. Where it is not practical to visit the plant, samples for both quality and size shall be taken from different parts of the car or boat during unloading. It is recommended that separate samples be taken from the top, middle and bottom of car or boat. These separate samples shall be well mixed in a composite sample and the sample for test obtained by the quartering method. **Sampling at Delivery.**

C. Quantity of Sand and Gravel Samples.

19. Samples of run of bank (where the sand and gravel are combined) shall consist of at least 100 lb. of material where the gravel content is 50 per cent or more of the whole. If the gravel is less in percentage, the sample shall be increased in proportion. For example, where the gravel percentage is 25 per cent of the whole the sample should contain 200 lb. **Run of Bank.**

Sand. 20. Samples of sand shall contain at least 20 lb. of material.

Gravel. 21. Samples of gravel shall contain at least 50 lb. of material.

Sample for Concrete Test. 22. The sample for concrete test will depend on the kind of tests to be made and the number of specimens necessary.

SAMPLING OF MISCELLANEOUS MATERIALS

23. Samples of slag sand, screenings, mine tailings, and all other materials used as a substitute for sand and gravel or broken stone, shall be inspected in the same manner and samples taken in the same way as the materials for which they are substituted.

SAMPLING OF STONE BLOCK

Where Sampled. 24. Samples shall be taken either at the quarry or from cars or boats as directed by the engineer. They shall be representative of the block which it is proposed to use and no sample shall include blocks that would be rejected by visual inspection.

Size of Sample. 25. The sample shall consist of at least six blocks and the bedding plane shall be marked on at least two of these.

GENERAL DIRECTIONS FOR SHIPPING AND MARKING SAMPLES

Stone and Slag. 26. Samples of ledge stone, crushed stone and slag shall be shipped in a secure box or bag.

Stone Block. 27. Samples of stone block shall be securely crated.

Gravel, Sand, Etc. 28. Samples of run of bank gravel, sand screenings, and other fine material, shall be shipped in a tight box or closely woven bag so there shall be no loss of the finer particles.

Marking. 29. Each sample or separate container shall be accompanied by a card or regular form, preferably in the container, giving the following information: By whom taken, official title or rank of the sampler; by whom submitted; source of supply; proposed use for the material; and in case of commercial supplies, daily production; geographic location, shipping facilities (name of railroad, canal or river, or other common carrier); and price of the material.

STANDARD METHOD OF MECHANICAL ANALYSIS OF SAND OR OTHER FINE HIGHWAY MATERIAL, EXCEPT FINE AGGREGATES USED IN CEMENT CONCRETE[1]

Serial Designation: D 7 – 27

This method is issued under the fixed designation D 7; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1911; REVISED, 1916, 1918, 1927.

Sampling. 1. A representative test sample of the aggregate weighing 50 g. shall be taken.

Treatment of Sample. 2. The sample shall be dried to constant weight at a temperature not exceeding 110° C. (230° F.).

Procedure. 3. (*a*) The sample shall be passed through each of the standard sieves specified in Table I.[2]

TABLE I.—REQUIREMENTS FOR SIEVE OPENINGS AND WIRE DIAMETERS WITH PERMISSIBLE VARIATIONS.[2]

Mesh Designation, U. S. Standard Sieve Series Number	Sieve Opening		Wire Diameter		Tolerance in Average Opening, per cent	Tolerance on Wire Diameter, per cent		Tolerance in Maximum Opening, per cent
	mm.	in.	mm.	in.		Under	Over	
10	2.00	0.0787	0.76	0.0299	±3	15	30	10
20	0.84	0.0331	0.42	0.0165	±5	15	30	25
30	0.59	0.0232	0.33	0.0130	±5	15	30	25
40	0.42	0.0165	0.25	0.0098	±5	15	30	25
50	0.297	0.0117	0.188	0.0074	±6	15	35	40
80	0.177	0.0070	0.119	0.0047	±6	15	35	40
100	0.149	0.0059	0.102	0.0040	±6	15	35	40
200	0.074	0.0029	0.053	0.0021	±8	15	35	60

NOTE.—The order in which the sieves are to be used in the process of sieving is immaterial and shall be left optional; but in reporting results the order in which the sieves have been used shall be stated.

(*b*) The percentage by weight retained on each sieve shall be determined and the sieving on each sieve shall be continued until

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] The dimensions and tolerances for these sieves conform to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, see p. 917.

less than 1 per cent of the weight retained on each sieve shall pass through the sieve during the last minute of sieving.

Report. 4. The percentages in mechanical analysis shall be reported in the following manner:

	PER CENT
Passing the No. 200 sieve.....	.....
Passing the No. 100 sieve and retained on the No. 200 sieve...	.....
Passing the No. 80 sieve and retained on the No. 100 sieve....	.....
Passing the No. 50 sieve and retained on the No. 80 sieve.....	.....
.....	.....
.....	.....
.....	.....
	100.00

STANDARD METHOD OF MECHANICAL ANALYSIS OF BROKEN STONE OR BROKEN SLAG, EXCEPT AGGREGATES USED IN CEMENT CONCRETE[1]

Serial Designation: D 18 – 16

This method is issued under the fixed designation D 18; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1916.

The method shall consist of (1) drying at not over 110° C. (230° F.) to a constant weight a sample weighing in pounds six times the diameter in inches of the largest holes required; (2) passing the sample through such of the following size screens having circular openings as are required or called for by the specifications, screens to be used in the order named: 8.89 cm. (3½ in.), 7.62 cm. (3 in.), 6.35 cm. (2½ in.), 5.08 cm. (2 in.), 3.81 cm. (1½ in.), 3.18 cm. (1¼ in.), 2.54 cm. (1 in.), 1.90 cm. (¾ in.), 1.27 cm. (½ in.), and 0.64 cm. (¼ in.); (3) determining the percentage by weight retained on each screen; and (4) recording the mechanical analysis in the following manner:

	PER CENT
Passing 0.64-cm. (¼-in.) screen	----------
Passing 1.27-cm. (½-in.) screen and retained on a 0.64-cm. (¼-in.) screen	----------
Passing 1.90-cm. (¾-in.) screen and retained on a 1.27-cm. (½-in.) screen	----------
Passing 2.54-cm. (1-in.) screen and retained on a 1.90-cm. (¾-in.) screen	----------
.....	----------
.....	----------
.....	----------
	100.00

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

STANDARD METHOD OF MECHANICAL ANALYSIS OF MIXTURES OF SAND OR OTHER FINE MATERIAL WITH BROKEN STONE OR BROKEN SLAG, EXCEPT AGGREGATES USED IN CEMENT CONCRETE[1]

Serial Designation: D 19 – 16

This method is issued under the fixed designation D 19; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1916.

The method shall consist of (1) drying at not over 110° C. (230° F.) to a constant weight a sample weighing in pounds six times the diameter in inches of the largest holes required; (2) separating the sample by the use of a screen having circular openings 0.64 cm. ($\frac{1}{4}$ in.) in diameter; (3) examining the portion retained on the screen in accordance with the Standard Method of Mechanical Analysis of Broken Stone or Broken Slag, Except Aggregates Used in Cement Concrete (Serial Designation: D 18) of the American Society for Testing Materials;[2] (4) examining the portion passing this screen in accordance with the Standard Method of Mechanical Analysis of Sand or Other Fine Highway Material, Except Fine Aggregates Used in Cement Concrete (Serial Designation D 7) of the American Society for Testing Materials;[3] and (5) recording the mechanical analysis in the following manner:

	PER CENT
Passing a No. 200 sieve	----------
Passing a No. 100 sieve and retained on a No. 200 sieve	----------
Passing a No. 80 sieve and retained on a No. 100 sieve	----------
.....	----------
Passing a No. 10 sieve and retained on a No. 20 sieve	----------
Passing 0.64-cm. ($\frac{1}{4}$-in.) screen and retained on a No. 10 sieve	----------
Passing 1.27-cm. ($\frac{1}{2}$-in.) screen and retained on a 0.64-cm. ($\frac{1}{4}$-in.) screen	----------
Passing 1.90-cm. ($\frac{3}{4}$-in.) screen and retained on a 1.27-cm. ($\frac{1}{2}$-in.) screen	----------
.....	----------
.....	----------
	100.00

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] See p. 481. [3] See p. 479.

STANDARD FORM

OF

SPECIFICATIONS FOR CERTAIN COMMERCIAL GRADES OF BROKEN STONE[1]

Serial Designation: D 35 – 18

This form of specifications is issued under the fixed designation D 35; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1916; ADOPTED, 1918.

The broken stone shall consist of one product of the operation of a stone-crushing and screening plant without recombining or mixing, and shall conform to the following mechanical analysis using laboratory screens:

Passing screen having smallest holes selected	From	to	per cent
Passing screen having next to largest holes selected	From	to	"
Passing screen having largest holes selected	From	to	"
Total passing screen having largest holes selected	100		"

Example.—The broken stone shall consist of one product of the operation of a stone-crushing and screening plant without recombining or mixing, and shall conform to the following mechanical analysis using laboratory screens:

Passing ¼-in. screen	3 to 10	per cent
Passing 1-in. screen and retained on ¼-in. screen	80 to 95	"
Passing 1¼-in. screen and retained on 1-in. screen	2 to 10	"
Total passing 1¼-in. screen	100	"

or the analysis may be expressed as follows.

Total passing ¼-in. screen	3 to 10	per cent
Total passing 1-in. "	90 to 98	"
Total passing 1¼-in. "	100	"

[1] Under the standardization procedure of the Society, this form of specifications is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

STANDARD METHOD OF TEST
FOR
THE DETERMINATION OF BITUMEN[1]

Serial Designation: D 4 - 27

This method is issued under the fixed designation D 4; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1911; REISSUED AS TENTATIVE, 1923; ADOPTED IN AMENDED FORM, 1927.

Scope.

1. Bitumen may usually be expeditiously and accurately determined by Method No. 1, Section 5. In the case of certain native asphalts containing finely divided mineral matter, the mineral residue is not easily retained by the filter and it is necessary to resort to Method No. 2, Section 6, in order to obtain accurate results. Method No. 2 shall not be used unless the filter clogs unduly or unless the mineral matter passing through the filter exceeds 0.5 per cent. In every case the report shall indicate whether Method No. 1 or Method No. 2 has been employed.

APPARATUS

Apparatus.

2. The apparatus shall consist of the following:

(*a*) Gooch crucible, approximately 4.4 cm. in width at the top, tapering to 3.6 cm. at the bottom, with a depth of 2.5 cm.;

(*b*) Filtering flask;

(*c*) Rubber stopper;

(*d*) Filter tube;

(*e*) Section of rubber tubing to hold the Gooch crucible on the filter tube;

(*f*) Asbestos (amphibole) cut in pieces not exceeding 1 cm. in length, shredded and shaken up with water;

(*g*) Two 150-cc. Erlenmeyer flasks;

(*h*) Bunsen burner;

(*i*) Drying oven;

(*j*) Suction pump;

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

(*k*) Rubber tubing;
(*l*) Analytical balance;
(*m*) Desiccator.

PREPARATION OF SAMPLE

Preparation of Sample.

3. The sample shall be representative and if it contains more than 2 per cent of water it shall be dehydrated by distillation in a copper still in accordance with the Tentative Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (Serial Designation: D 20 – 27 T) of the American Society for Testing Materials,[1] the water-free distillate being returned to the residue. If the material is hard and brittle, it may be ground and dried at a temperature below the temperature of volatilization of the material.

PROCEDURE

Preparation of Gooch Crucible.

4. The Gooch crucible shall be set in the filter tube inserted in the stopper of the filtering flask. The flask shall be connected with the suction pump. Before suction shall be applied, the crucible shall be filled with asbestos suspended in water which shall be allowed to partly settle in the crucible. A light suction shall be applied to draw off the water, leaving a firm mat of asbestos in the crucible. More suspended asbestos shall be added and the process repeated until a felt is built up that barely transmits light. The felt shall then be thoroughly washed with water, dried in a drying oven, and ignited over a Bunsen burner. The crucible shall then be cooled in a desiccator and weighed.

(*a*) *Method No. 1*

Procedure No. 1.

5. An amount of material which shall contain approximately one gram of bitumen shall be weighed into a tared Erlenmeyer flask. One hundred cubic centimeters of chemically pure carbon disulfide shall be added to the flask in small portions with continued agitation until all lumps disappear and nothing adheres to the bottom. The flask shall be corked and set aside for fifteen minutes.

The Gooch crucible shall be set up again with the suction flask and the carbon disulfide solution carefully decanted through the asbestos felt, with or without light suction as may be found necessary. No sediment shall be allowed to go onto the filter. A small amount of carbon disulfide shall be used to wash down the sides of the flask and then the precipitate shall be brought onto the felt and the flask scrubbed with a feather if necessary to remove all precipitate. The

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 505.

contents of the crucible shall be washed with carbon disulfide until the washings are colorless. Suction shall be applied to the crucible to remove the carbon disulfide. The crucible shall be dried in the oven at 100 to 125° C. for twenty minutes, cooled in the desiccator and weighed.

In case insoluble matter adheres to the flask, the flask shall be dried and weighed and the increase in weight over the original weight shall be added to the weight of insoluble matter in the crucible.

The crucible shall be ignited at a red heat and after thorough ignition, cooled and weighed.

The weight of substance taken, minus the total weight of matter insoluble in carbon disulfide, is the total bitumen. The weight of material after ignition is ash.

(*b*) *Method No. 2*

Procedure No. 2.

6. Material shall be weighed out in the same way as in (*a*) Method No. 1 (Section 5) into a tared Erlenmeyer flask, and treated with 100 cc. of chemically pure carbon disulfide.

The flask shall be loosely corked and shaken at intervals until all large particles of material have been broken down. The flask shall be left undisturbed for 48 hours. The solution shall be decanted into a similar tared flask, care being taken to disturb as little of the residue as possible. The first flask shall be treated again with fresh carbon disulfide as before and left undisturbed for 48 hours. The solution from the second flask shall then be carefully decanted upon the Gooch crucible without use of vacuum, and this shall be followed by the solution from the first flask. The filter shall be washed with fresh carbon disulfide. The residue remaining in each flask shall be shaken again with fresh carbon disulfide and allowed to settle for 24 hours. The solution from both flasks shall then be decanted through the filter and the residues remaining in the flasks shall be washed again with carbon disulfide, the solution decanted and the process repeated until the washings are practically colorless. The temperature shall be maintained between 20 and 25° C.

The crucible and both flasks shall be dried at 100 to 125° C. and weighed. The filtrate containing the bitumen shall be evaporated, the bituminous residue burned, and the weight of the ash thus obtained added to that of the residue in the two flasks and the crucible. The sum of these weights deducted from the weight of substance taken is the weight of bitumen.

STANDARD METHOD OF TEST

FOR

THE DETERMINATION OF PROPORTION OF BITUMEN SOLUBLE IN CARBON TETRACHLORIDE[1]

Serial Designation: D 165 - 27

This method is issued under the fixed designation D 165; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED IN AMENDED FORM, 1927.

APPARATUS

Apparatus.

1. The apparatus shall consist of the following:

(*a*) Gooch Crucible, approximately 4.4 cm. in width at the top, tapering to 3.6 cm. at the bottom, with a depth of 2.5 cm.

(*b*) Filtering flask;

(*c*) Rubber stopper;

(*d*) Filter tube;

(*e*) Section of rubber tubing to hold the Gooch crucible on the filter tube;

(*f*) Asbestos (amphibole) cut in pieces not exceeding 1 cm. in length, shredded and shaken up with water;

(*g*) Two 150-cc. Erlenmeyer flasks;

(*h*) Bunsen burner;

(*i*) Drying oven;

(*j*) Suction pump;

(*k*) Rubber tubing;

(*l*) Analytical balance;

(*m*) Desiccator.

PREPARATION OF SAMPLE

Preparation of Sample.

2. The sample shall be representative and if it contains more than 2 per cent of water it shall be dehydrated by distillation in a copper still in accordance with the Tentative Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (Serial Designation: D 20 - 27 T) of the American Society for Testing Materials,[2]

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 505.

and water-free distillate returned to the residue. If the material is hard and brittle, it may be ground and dried at a temperature below the temperature of volatilization of the material.

PROCEDURE

Preparation of Gooch Crucible.

3. The Gooch crucible shall be set in the filter tube inserted in the stopper of the filtering flask. The flask shall be connected with the suction pump. Before suction is applied, the crucible shall be filled with asbestos suspended in water which shall be allowed to partly settle in the crucible. A light suction shall be applied to draw off the water, leaving a firm mat of asbestos in the crucible. More suspended asbestos shall be added and the process repeated until a felt is built up that barely transmits light. The felt shall then be thoroughly washed with water, dried in a drying oven, and ignited over a Bunsen burner. The crucible then shall be cooled in a desiccator and weighed.

Procedure.

4. An amount of material which shall contain approximately one gram of bitumen shall be weighed into a tared Erlenmeyer flask. One hundred cubic centimeters of chemically pure carbon tetrachloride shall be added to the flask in small portions with continued agitation until all lumps disappear and nothing adheres to the bottom. The flask shall be corked and set aside in subdued light for at least 12 hours.

The Gooch crucible shall be set up again with the suction flask and the carbon tetrachloride solution carefully decanted through the asbestos felt, with or without light suction as may be found necessary. No sediment shall be allowed to go onto the filter. A small amount of carbon tetrachloride shall be used to wash down the sides of the flask and then the precipitate shall be brought onto the felt and the flask scrubbed with a feather if necessary to remove all precipitate. The contents of the crucible shall be washed with carbon tetrachloride until the washings are colorless. Suction shall be applied to the crucible to remove the carbon tetrachloride. The crucible shall be dried in the oven at 100 to 125° C. for twenty minutes, cooled in the desiccator, and weighed.

In case insoluble matter adheres to the flask, the flask shall be dried and weighed and the increase in weight over the original weight shall be added to the weight of insoluble matter in the crucible.

The proportion of bitumen soluble in carbon tetrachloride shall be reported on the basis of total bitumen taken as 100:

$$\text{Proportion of Bitumen Soluble in Carbon Tetrachloride.} = \frac{\text{Bitumen Soluble in Carbon Tetrachloride}}{\text{Total Bitumen}}$$

STANDARD METHOD OF TEST FOR LOSS ON HEATING OF OIL AND ASPHALTIC COMPOUNDS[1]

Serial Designation: D 6 – 27

This method is issued under the fixed designation D 6; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1911; REVISED, 1916, 1920, 1927.

Scope.

1. This test covers the determination of the loss in weight (exclusive of water) of oil and asphaltic compounds when heated as hereinafter prescribed. The material under examination shall, therefore, first be tested for water and if water is found to be present, it shall be removed by suitable methods of dehydration before the material is subjected to the loss on heating test; or another sample shall be obtained which is free from water.

APPARATUS

Oven.

2. The oven[1] may be either circular or rectangular in form and may be heated by either gas or electricity. Its interior dimensions shall be as follows: height, not less than 40.64 cm. (16 in.); width and depth or diameter, at least 5.08 cm. (2 in.) greater than the diameter of the revolving shelf.

It shall be well ventilated and shall be fitted with a window in the upper half of the door, so placed and of sufficient size to permit the accurate reading of the thermometer without opening the door. It shall also be provided with a perforated circular shelf preferably of approximately 24.8 cm. (9.75 in.) in diameter. This shelf shall be placed in the center of the oven and shall be suspended by a vertical shaft and provided with mechanical means for rotating it at the rate of 5 to 6 revolutions per minute. It shall be provided with recesses equidistant from the central shaft in which the tins containing the samples are to be placed. (A recommended form of aluminum shelf is shown in Fig. 1.)

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

The committee calls attention to the fact that the question of size of oven is now under consideration and that a possible revision of this standard may be expected.

Thermometer.

3. The thermometer shall conform to the following requirements. These specifications cover a special thermometer graduated in Centigrade degrees, the range being 155 to 170° C.

TYPE: Etched stem, glass.
LIQUID: Mercury.
RANGE AND SUBDIVISION: 155 to 170° C. in 0.5° C.
TOTAL LENGTH: 150 to 154 mm. (5.90 to 6.06 in.).
STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).
BULB: Corning normal or equally suitable thermometric glass. Length, 10 to 15 mm. (0.39 to 0.59 in.). Diameter, 5.0 to 6.0 mm. (0.196 to 0.236 in.).
DISTANCE TO 0° C. LINE FROM BOTTOM OF BULB: 33 to 38 mm. (1.30 to 1.49 in.).
DISTANCE TO 155° C. LINE FROM BOTTOM OF BULB: 70 to 80 mm. (2.75 to 3.15 in.).

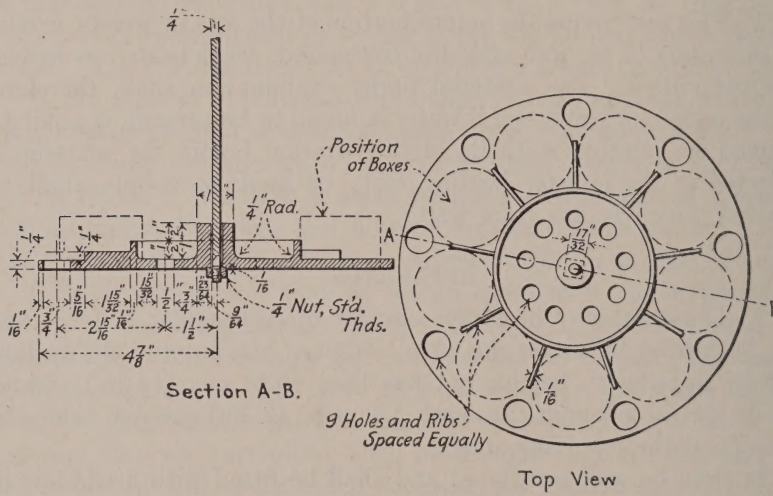


FIG. 1.—Aluminum Shelf.

DISTANCE TO 170° C. LINE FROM TOP OF THERMOMETER: 20 to 30 mm. (0.79 to 1.18 in.).
CONTRACTION CHAMBER: To be long narrow type, top to be not more than 65 mm. (2.56 in.) above bottom of the bulb.
EXPANSION CHAMBER: To permit heating the thermometer at least 25° C. above the highest temperature on the scale.
FILLING ABOVE MERCURY: Nitrogen gas.
TOP FINISH: Glass ring.
GRADUATION: All lines and figures clear cut and distinct. Each whole degree centigrade line to be longer than the remaining lines. Graduations are to be numbered at 0, 155, 163 and 170° C.
IMMERSION: Total.
SPECIAL MARKING: "A.S.T.M. Loss on Heat," a serial number and the manufacturer's name or trade mark shall be etched on the stem.

SCALE ERROR: The error at any point of the scale, when the thermometer is standardized as provided below shall not exceed 0.5° C.

STANDARDIZATION: The thermometer shall be standardized at three points including 163° C. for total immersion.

CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. Loss on Heat, 155 to 170° C."

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

Container.

4. The container in which the sample is to be tested shall be of tin, cylindrical in shape, and shall have a flat bottom. Its inside dimensions shall be substantially as follows: diameter, 55 mm. (2.17 in.); depth, 35 mm. (1.38 in.). (A 3-oz. Gill style ointment box, deep pattern, fulfills these requirements.)

PREPARATION OF SAMPLE

Preparation of Sample.

5. The sample as received shall be thoroughly stirred and agitated, warming, if necessary, to insure a complete mixture before the portion for analysis is removed.

PROCEDURE

Procedure.

6. Fifty grams of the water-free material to be tested shall be weighed into a tared container conforming to the requirements of Section 4. The oven shall be brought to a temperature of 163° C. (325° F.), and the tin box containing the sample placed in one of the recesses of the revolving shelf. The thermometer shall be immersed for the depth of its bulb in a separate 50-g. sample of the material under test, placed in a similar container, and shall be conveniently suspended from the vertical shaft. This sample shall rest in one of the recesses upon the same shelf and revolve with the sample or samples under test. The oven shall then be closed and the shelf rotated 5 to 6 revolutions per minute during the entire test. The temperature shall be maintained at 163° C. (325° F.) for 5 hours, then the sample shall be removed from the oven, cooled and weighed, and the loss due to volatilization calculated.

Permissible Variation in Temperature.

7. During the 5-hour period the temperature shall not vary more than 1° C. All tests showing a greater variation in temperature shall be rejected.

Precautions.

8. Under ordinary circumstances a number of samples having about the same degree of volatility may be tested at the same time. Samples varying greatly in volatility should be tested separately. Where extreme accuracy is required not more than one material should be tested at one time and duplicate samples of it should be placed simultaneously in the oven. Such duplicates shall check within the limits of accuracy given in Section 9. Results obtained on samples showing evidences of foaming during the test shall be rejected.

ACCURACY

Accuracy.

9. Up to 5 per cent loss in weight the results obtained may be considered as correct within 0.5. Above 5 per cent loss in weight the numerical limit of error increases 0.01 for every 0.5 per cent increase in loss by volatilization as follows:

VOLATILIZATION LOSS, PER CENT	NUMERICAL CORRECTION	TRUE VOLATILIZATION LOSS, PER CENT
5.0	±0.50	4.50 to 5.50
5.5	±0.51	4.91 " 6.01
6.0	±0.52	5.48 " 6.52
10.0	±0.60	9.40 " 10.60
15.0	±0.70	14.30 " 15.70
25.0	±0.90	24.10 " 25.90
40.0	±1.20	38.80 " 41.20

NOTE.—If additional periods of heating are desired, it is recommended that they be made in successive increments of 5 hours each.

When the penetration of the sample after heating is required, melt the residue in the container at the lowest possible temperature and thoroughly mix by stirring, taking care to avoid incorporating air bubbles in the mass. Then bring it to the standard temperature and test as prescribed under the directions for the Standard Method of Test for Penetration of Bituminous Materials (Serial Designation: D 5) of the American Society for Testing Materials.[1]

[1] See p. 493.

STANDARD METHOD OF TEST
FOR
PENETRATION OF BITUMINOUS MATERIALS[1]

Serial Designation: D 5 – 25

This method is issued under the fixed designation D 5; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1911; REVISED, 1916, 1921, 1925.

DEFINITION

Definition of Penetration.

1. Penetration is defined as the consistency of a bituminous material, expressed as the distance that a standard needle vertically penetrates a sample of the material under known conditions of loading, time and temperature. Where the conditions of test are not specifically mentioned, the load, time and temperature are understood to be 100 g., 5 seconds, 25° C. (77° F.), respectively, and the units of penetration to indicate hundredths of a centimeter.

APPARATUS

Container.

2. The container for holding the material to be tested shall be a flat-bottom, cylindrical dish, 55 mm. ($2\frac{3}{16}$ in.) in diameter and 35 mm. ($1\frac{3}{8}$ in.) deep.[2]

Needle.

3. The needle (Fig. 1) for this test shall be made from a cylindrical steel rod approximately 50.8 mm. (2 in.) long, and having a diameter of 1.00 to 1.02 mm. This shall be symmetrically tapered at one end to a cone approximately 6.35 mm. ($\frac{1}{4}$ in.) in height and whose angle shall be within the range of 8° 40′ and 9° 40′. After tapering, the point shall be "blunted" by grinding off to a truncated cone, the smaller base of which shall be from 0.14 to 0.16 mm. in diameter. The finished needle shall be hardened and highly polished.

NOTE.—The U. S. Bureau of Standards will measure and certify the accuracy of penetration needles in accordance with the tolerances given.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] This requirement is fulfilled by the American Can Company's Gill style ointment box, deep pattern, 3 oz. capacity.

Water Bath.

4. The water bath shall be maintained at a temperature not varying more than 0.1° C. from 25° C. (77° F.). The volume of water shall be not less than 10 liters and the sample shall be immersed to a depth of not less than 10 cm. (4 in.) and shall be supported on a perforated shelf not less than 5 cm. (2 in.) from the bottom of the bath.

Apparatus for Penetration.

5. Any apparatus which will allow the needle to penetrate without appreciable friction, and which is accurately calibrated to yield results in accordance with the definition of penetration, will be acceptable.

Transfer Dish for Container.

6. The transfer dish for container shall be a small dish or tray of such capacity as will insure complete immersion of the container during the test. It shall be provided with some means which will insure a firm bearing and prevent rocking of the container.

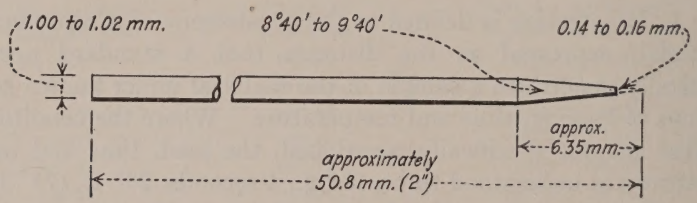


FIG. 1.—Needle for Penetration Test.

PREPARATION OF SAMPLE

Preparation of Sample.

7. The sample shall be completely melted at the lowest possible temperature and stirred thoroughly until it is homogeneous and free from air bubbles. It shall then be poured into the sample container to a depth of not less than 15 mm. ($\frac{5}{8}$ in.). The sample shall be protected from dust and allowed to cool in an atmosphere not lower than 18° C. (65° F.) for one hour. It shall then be placed in the water bath along with the transfer dish and allowed to remain one hour.

PROCEDURE

Procedure.

8. (*a*) In making the test the sample shall be placed in the transfer dish filled with water from the water bath of sufficient depth to completely cover the container. The transfer dish containing the sample shall then be placed upon the stand of the penetration machine. The needle loaded with specified weight, shall be adjusted to make contact with the surface of the sample. This may be accomplished by making contact of

the actual needle point with its image reflected by the surface of the sample from a properly placed source of light. Either the reading of the dial shall then be noted or the needle brought to zero. The needle is then released for the specified period of time, after which the penetration machine is adjusted to measure the distance penetrated.

At least three tests shall be made at points on the surface of the sample not less than 1 cm. ($\frac{3}{8}$ in.) from the side of the container and not less than 1 cm. ($\frac{3}{8}$ in.) apart. After each test the sample and transfer dish shall be returned to the water bath and the needle shall be carefully wiped toward its point with a clean, dry cloth to remove all adhering bitumen. The reported penetration shall be the average of at least three tests whose values shall not differ more than four points between maximum and minimum.

(*b*) When desirable to vary the temperature, time and weight, and, in order to provide for a uniform method of reporting results when variations are made, the samples shall be melted and cooled in air as above directed. They shall then be immersed in water or brine, as the case may require, for one hour at the temperature desired. The following combinations are suggested:

At 0° C. (32° F.) 200-g. weight, 60 seconds.
At 46.1° C. (115° F.) 50-g. weight, 5 seconds.

STANDARD METHOD OF TEST
FOR
SOFTENING POINT OF BITUMINOUS MATERIALS[1]
(RING-AND-BALL METHOD)

Serial Designation: D 36 - 26

This method is issued under the fixed designation D 36; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1916; ADOPTED IN AMENDED FORM, 1919; REVISED, 1921, 1924, 1926.

Softening Point.

1. The softening of bituminous materials generally takes place at no definite moment or temperature. As the temperature rises, they gradually and imperceptibly change from a brittle or exceedingly thick and slow flowing material to a softer and less viscous liquid. For this reason the determination of the softening point must be made by a fixed, arbitrary and closely defined method if the results obtained are to be comparable.

APPARATUS

Ring.

2. The apparatus shall consist of the following:

(*a*) A brass ring 15.875 mm. ($\frac{5}{8}$ in.) in inside diameter and 6.35 mm. ($\frac{1}{4}$ in.) deep; thickness of wall, 2.38 mm. ($\frac{3}{32}$ in.); permissible variation on inside diameter and thickness of ring 0.25 mm. (0.01 in.). This ring shall be attached in a convenient manner to a No. 13 B. & S. gage brass wire (diameter 1.83 mm. = 0.072 in.). See Fig. 1.

Ball.

(*b*) A steel ball 9.53 mm. ($\frac{3}{8}$ in.) in diameter weighing between 3.45 and 3.55 g.

Container.

(*c*) A glass vessel, capable of being heated, not less than 8.5 cm. (3.34 in.) in diameter and measuring 10.5 cm. (4.13 in.) in depth from the bottom of the flare. (A 600-cc. beaker, low form, meets this requirement.)

Thermometer.

(*d*) A thermometer which shall conform to the following requirements. These specifications cover a special thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being −2 to +80° C. or +30 to +180° F., respectively.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

TYPE: Etched stem, glass.
LIQUID: Mercury.
RANGE AND SUBDIVISION: −2 to +80° C. in 0.2° C. or +30 to +180° F. in 0.5° F.
TOTAL LENGTH: 378 to 384 mm. (14.88 to 15.12 in.).
STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).

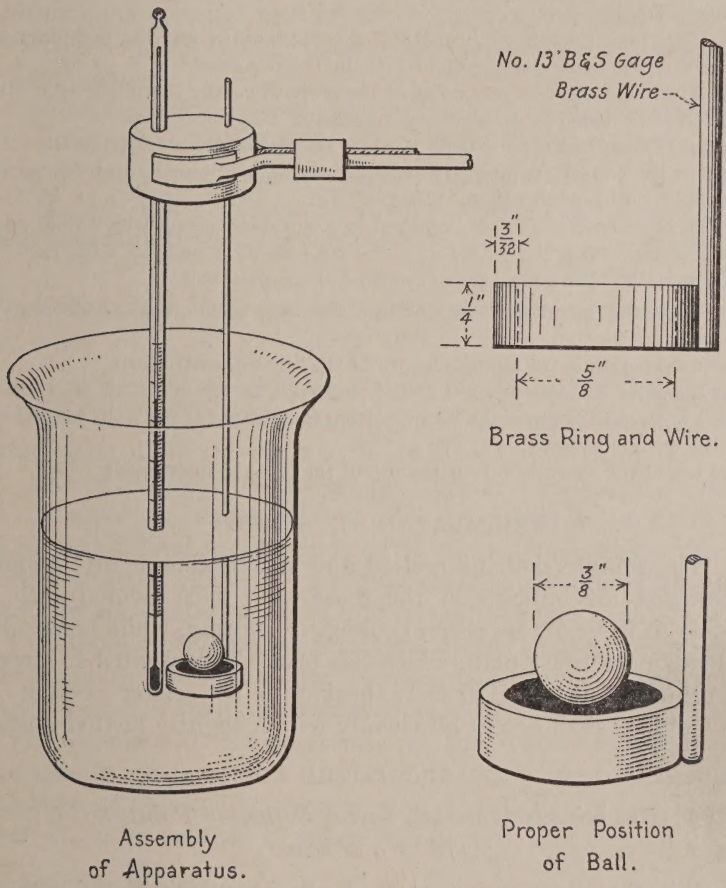


FIG. 1.—Apparatus for Ring-and-Ball Method.

BULB: Corning normal or equally suitable thermometric glass.
Length, 9 to 14 mm. (0.35 to 0.55 in.).
Diameter, 4.5 to 5.5 mm. (0.18 to 0.22 in.).
DISTANCE TO 0° C. OR 32° F. LINE FROM BOTTOM OF BULB: 75 to 90 mm. (2.95 to 3.54 in.).
DISTANCE TO 80° C. OR 176° F. LINE FROM TOP OF THERMOMETER: 30 to 45 mm. (1.18 to 1.77 in.).

EXPANSION CHAMBER: To permit heating the thermometer at least 50° C. (90° F.) above highest temperature on scale.

FILLING ABOVE MERCURY: Nitrogen gas.

TOP FINISH: Glass Ring.

GRADUATION: All lines, figures, and letters clear cut and distinct. Each whole degree Centigrade or Fahrenheit line to be longer than the remaining lines. Graduations to be numbered at each multiple of 2° C. or 5° F.

IMMERSION: Total.

SPECIAL MARKING: "A.S.T.M. Low S.P.," a serial number and the manufacturer's name or trade mark shall be etched on the thermometer.

SCALE ERROR: The error at any point of the scale when the thermometer is standardized as provided below shall not exceed 0.2° C. or 0.4° F.

STANDARDIZATION: The thermometer shall be standardized immersed in the testing bath to the top of the mercury column, at the ice point and at temperature intervals of approximately 20° C. or 40° F.

CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. Low S.P., −2 to +80° C.," or "A.S.T.M. Low S.P., +30 to +180° F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

PREPARATION OF SAMPLE

Preparation of Sample.

3. The sample shall be melted and stirred thoroughly, avoiding incorporating air bubbles in the mass, and then poured into the ring so as to leave an excess on cooling. The ring, while being filled, should rest on a brass plate which has been amalgamated to prevent the bituminous material from adhering to it. After cooling, the excess material shall be cut off cleanly with a slightly heated knife.

PROCEDURE

(*A*) *Bituminous Materials having Softening Points 80° C. (176° F.) or below*

Assembling.

4. Fill the glass vessel to a depth of substantially 8.25 cm. (3.25 in.) with freshly boiled, distilled water at 5° C. (41° F.). Suspend the ring containing the sample in the water so that the lower surface of the filled ring is exactly 2.54 cm. (1 in.) above the bottom of the glass vessel and its upper surface is 5.08 cm. (2 in.) below the surface of the water. Place the ball in the water but not on the specimen. Suspend the thermometer so that the bottom of the bulb is level with the bottom of the ring and within 0.635 cm. ($\frac{1}{4}$ in.), but not touching the ring. Maintain the temperature of the water at 5° C.

(41° F.) for 15 minutes. With suitable forceps, place the ball in the center of the upper surface of the bitumen in the ring, thus completing the assembly as in Fig. 1.

Heating. 5. Apply the heat in such a manner that the temperature of the water is raised 5° C. (9° F.) each minute.

Softening Point. 6. The temperature recorded by the thermometer at the instant the bituminous material touches the bottom of the glass vessel shall be reported as the softening point. No correction shall be made for emergent stem.

Permissible Variation in Rise of Temperature. 7. The rate of rise of temperature shall be uniform and shall not be averaged over the period of the test. The maximum permissible variation for any minute period after the first three shall be ±0.5° C. (0.9° F.). All tests in which the rate of rise in temperature exceeds these limits shall be rejected.

(*B*) *Bituminous Materials having Softening Points above 80° C. (176° F.)*

Modifications for Hard Materials. 8. The same method as given under (*A*) shall be employed except that U. S. P. glycerin shall be used instead of water, and the starting point of the glycerin bath shall be 32° C. (89.6° F.). The bath shall be brought to this temperature and thoroughly agitated, then the apparatus and specimens shall be placed in the bath which shall be maintained under agitation at the starting temperature for 15 minutes, after which the assembly shall be completed by placing the ball on the center of the specimen and the test carried on as in (*A*). In applying the heat, the ring apparatus shall be placed off the center of the container and the burner placed midway between the center and edge of the beaker away from the specimen.

Thermometer The thermometer shall conform to the following requirements. These specifications cover a special thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being 30 to 160° C. or 85 to 320° F., respectively.

TYPE: Etched stem, glass.
LIQUID: Mercury.
RANGE AND SUBDIVISION: 30 to 160° C. in 0.5° C. or 85 to 320° F. in 1° F.
TOTAL LENGTH: 378 to 384 mm. (14.88 to 15.12 in.).
STEM: Plain front, enamel back, suitable thermometer tubing.
Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).
BULB: Corning normal or equally suitable thermometric glass.
Length, 9 to 14 mm. (0.35 to 0.55 in.).
Diameter, 4.5 to 5.5 mm. (0.18 to 0.22 in.).
DISTANCE TO 30° C. OR 86° F. LINE FROM BOTTOM OF BULB: 75 to 90 mm. (2.95 to 3.54 in.).

DISTANCE TO 160° C. OR 320° F. LINE FROM TOP OF THERMOMETER: 30 to 45 mm. (1.18 to 1.77 in.).

EXPANSION CHAMBER: To permit heating the thermometer at least 50° C. (90° F.) above the highest temperature on the scale.

FILLING ABOVE MERCURY: Nitrogen gas.

TOP FINISH: Glass ring.

GRADUATION: All lines, figures, and letters clear cut and distinct. Each whole degree Centigrade line or the first and each succeeding 5° F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 5° C. or 10° F.

IMMERSION: Total.

SPECIAL MARKING: "A.S.T.M. High S.P.," a serial number and the manufacturer's name or trade mark shall be etched on the thermometer.

SCALE ERROR: The error at any point of the scale when the thermometer is standardized as provided below shall not exceed 0.3° C. or 0.5° F.

STANDARDIZATION: The thermometer shall be standardized, immersed in the testing bath to the top of the mercury column, at intervals of approximately 40° C. or 70° F.

CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. High S.P., 30 to 160° C.," or "A.S.T.M. High S.P., 85 to 320° F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

PRECAUTIONS

Precautions.

9. The use of freshly boiled distilled water is essential, as otherwise air bubbles may form on the specimen and affect the accuracy of the results. Rigid adherence to the prescribed rate of heating is absolutely essential in order to secure accuracy of results.

A sheet of paper placed on the bottom of the glass vessel and conveniently weighted will prevent the bituminous material from sticking to the glass vessel, thereby saving considerable time and trouble in cleaning.

ACCURACY

Accuracy.

10. The limit of accuracy of the test is ±0.5° C. (0.9° F.).

STANDARD METHOD OF TEST
FOR
SOFTENING POINT OF TAR PRODUCTS[1]
(CUBE-IN-WATER METHOD)

Serial Designation: D 61 - 24

This method is issued under the fixed designation D 61; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1919; ADOPTED IN AMENDED FORM, 1920; REVISED, 1924.

Scope.

1. (*a*) The softening of pitch takes place at no definite moment or temperature. As the temperature rises, pitch gradually and imperceptibly changes from a brittle or exceedingly thick and slow flowing material to a softer and less viscous liquid. For this reason the determination of the softening point must be made by a fixed, arbitrary and closely defined method if the results obtained are to be comparable.

(*b*) The methods herein described are not applicable to pitches having a melting point above 77° C. (170.6° F.).

(*c*) For the purpose of shortening the time required for testing, hard pitches having a softening point between 43 and 77° C. (109.4 and 170.6° F.) are cooled at 15.5° C. (60° F.) instead of at 4° C. (39.2° F.) as prescribed for soft pitches.

APPARATUS

2. The apparatus shall consist of the following:

Mold.

(*a*) A mold suitable for forming a 12.7-mm. ($\frac{1}{2}$-in.) cube of pitch. (A recommended type is shown in Fig. 1.)

Hook.

(*b*) An L-shaped right angled hook made of No. 12 B. & S. gage copper wire (diameter 2.05 mm. = 0.0808 in.) the foot of which shall be 2.54 cm. (1 in.) long. See Fig. 1.

Container.

(*c*) A glass vessel, capable of being heated, not less than 8.5 cm. (3.34 in.) in diameter and measuring 10.5 cm. (4.13 in.) in depth from the bottom of the flare. (A 600-cc. beaker, Griffin low form, meets this requirement.)

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

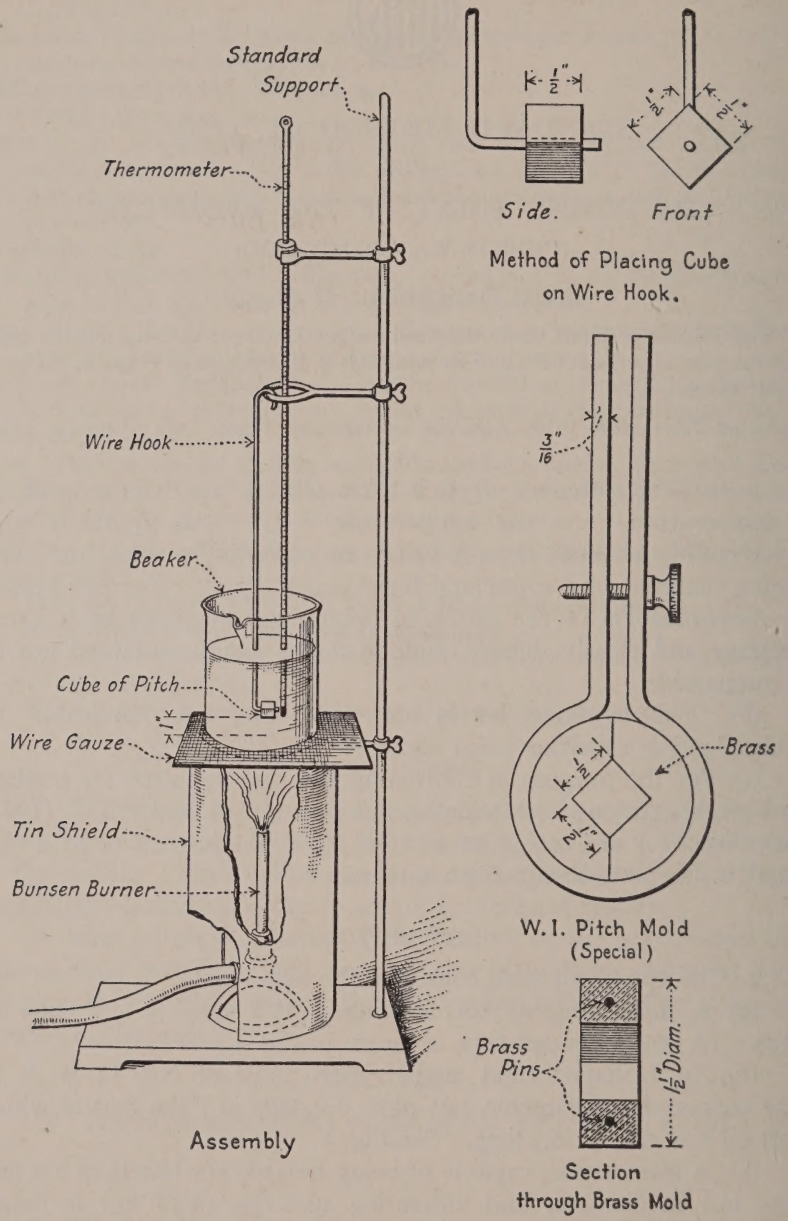


FIG. 1.—Apparatus for Cube-in-Water Method.

(*d*) A thermometer which shall conform to the following requirements. These specifications cover a special thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being −2 to +80° C. or +30 to +180° F., respectively. Thermometer.

TYPE: Etched stem, glass.

LIQUID: Mercury.

RANGE AND SUBDIVISION: −2 to +80° C. in 0.2° C. or +30 to +180° F. in 0.5° F.

TOTAL LENGTH: 378 to 384 mm. (14.88 to 15.12 in.).

STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).

BULB: Corning normal or equally suitable thermometric glass.
Length, 9 to 14 mm. (0.35 to 0.55 in.).
Diameter, 4.5 to 5.5 mm. (0.18 to 0.22 in.).

DISTANCE TO 0° C. OR 32° F. LINE FROM BOTTOM OF BULB: 75 to 90 mm. (2.95 to 3.54 in.).

DISTANCE TO 80° C. OR 176° F. LINE FROM TOP OF THERMOMETER: 30 to 45 mm. (1.18 to 1.77 in.).

EXPANSION CHAMBER: To permit heating the thermometer at least 50° C. (90° F.) above highest temperature on scale.

FILLING ABOVE MERCURY: Nitrogen gas.

TOP FINISH: Glass Ring.

GRADUATION: All lines, figures, and letters clear cut and distinct. Each whole degree Centigrade or Fahrenheit line to be longer than the remaining lines. Graduations to be numbered at each multiple of 2° C. or 5° F.

IMMERSION: Total.

SPECIAL MARKING: "A.S.T.M. Low S.P.," a serial number and the manufacturer's name or trade mark shall be etched on the thermometer.

SCALE ERROR: The error at any point of the scale when the thermometer is standardized as provided below shall not exceed 0.2° C. or 0.4° F.

STANDARDIZATION: The thermometer shall be standardized immersed in the testing bath to the top of the mercury column, at the ice point and at temperature intervals of approximately 20° C. or 40° F.

CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. Low S.P., −2 to +80° C." or "A.S.T.M. Low S.P., +30 to +180° F.", according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

PREPARATION OF SAMPLE

3. The pitch shall be formed into a 12.7-mm. ($\frac{1}{2}$-in.) cube, truly shaped and with sharp edges, either by melting and pouring, or softening and pressing, into a mold. In all cases an excess of pitch shall be used and the surplus material shall be cut off cleanly with a Preparation of Sample.

slightly heated knife. The harder pitches specified can ordinarily be molded at room temperature, the softer pitches in water at about 4° C. (39.2° F.). If they are melted, they should first be thoroughly stirred, avoiding incorporating air bubbles in the mass, and then poured into the mold so as to leave an excess on cooling. The mold should rest on a brass plate and the surface of the plate and the interior surfaces of the mold should be amalgamated to prevent the pitch from adhering to them.

PROCEDURE

(*A*) *Pitches having Softening Points between 43 and 77° C. (109.4 and 170.6° F.)*

Assembling.

4. Assemble the apparatus as shown in Fig. 1. Fill the glass vessel to a depth of substantially 9.5 cm. (3.75 in.) with freshly boiled, distilled water at 15.5° C. (60° F.). Place the cube of pitch on the wire as shown in Fig. 1 and suspend it in the water so that its lower edge is exactly 2.54 cm. (1 in.) above the bottom of the glass vessel and its upper edge is 5.08 cm. (2 in.) below the surface of the water. Allow it to remain in the water for 15 minutes before applying heat. Suspend the thermometer so that the bottom of the bulb is level with the bottom edge of the cube of pitch and within 0.635 cm. ($\frac{1}{4}$ in.), but not touching the cube.

Heating.

5. Apply the heat in such a manner that the temperature of the water is raised 5° C. (9° F.) each minute.

Softening Point.

6. The temperature recorded by the thermometer at the instant the pitch touches the bottom of the glass vessel shall be reported as the softening point. No correction shall be made for emergent stem.

Permissible Variation in Rise of Temperature.

7. The rate of rise of temperature shall be uniform and shall not be averaged over the period of the test. The maximum permissible variation for any minute period after the first three shall be $\pm 0.5°$ C. (0.9° F.). All tests in which the rate of rise in temperature exceeds these limits shall be rejected.

(*B*) *Pitches having Softening Points below 43° C. (109.4° F.)*

Modification for Soft Pitches.

8. Use the same method as given under (*A*), except that the water when placed in the glass vessel shall be at a temperature of 4° C. (39.2° F.). The cube shall be allowed to remain 15 minutes in this water before applying the heat.

PRECAUTIONS

Precautions.

9. The use of freshly distilled water is essential, as otherwise air bubbles may form on the cube and retard its sinking. Rigid

adherence to the prescribed rate of heating is absolutely essential in order to secure accuracy of results.

A sheet of paper placed on the bottom of the glass vessel and conveniently weighted will prevent the pitch from sticking to the glass vessel, thereby saving considerable time and trouble in cleaning.

ACCURACY

10. The limit of accuracy of the test is ±0.5° C. (0.9° F.). Accuracy.

STANDARD METHOD OF FLOAT TEST FOR BITUMINOUS MATERIALS[1]

Serial Designation: D 139 – 27

This method is issued under the fixed designation D 139; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED IN AMENDED FORM, 1927.

APPARATUS

Float.

1. The float (Fig. 1) shall be made of aluminum or aluminum alloy and shall be in accordance with the following requirements:

	MINIMUM.	NORMAL.	MAXIMUM.
Weight of float, g	37.70	37.90	38.10
Total height of float, mm	34.0	35.0	36.0
Height of rim above lower side of shoulder, mm	26.5	27.0	27.5
Thickness of shoulder, mm	1.3	1.4	1.5
Diameter of opening, mm	11.0	11.1	11.2

Collar.

2. The collar (Fig. 1) shall be made of brass and shall be in accordance with the following requirements:

	MINIMUM.	NORMAL.	MAXIMUM.
Weight of collar, g	9.60	9.80	10.00
Over-all height of collar, mm	22.3	22.5	22.7
Inside diameter at bottom, mm	12.72	12.82	12.92
Inside diameter at top, mm	9.65	9.70	9.75

The top of the collar shall screw up tightly against the lower side of the shoulder.

Assembly.

3. The assembled float and collar, with the collar filled flush with the bottom and weighted to a total weight of 53.2 g., shall float upon water with the rim 8.5 ±1.5 mm. above the surface of the water. Dimensions of the apparatus additional to those required above are given in Fig. 1.

Thermometer.

4. The thermometer shall conform to the following requirements. These specifications cover a special thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being −2 to +80° C. or +30 to +180° F., respectively.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

TYPE: Etched stem, glass.
LIQUID: Mercury.
RANGE AND SUBDIVISION: −2 to +80° C. in 0.2° C. or +30 to +180° F. in 0.5° F.
TOTAL LENGTH: 378 to 384 mm. (14.88 to 15.12 in.).

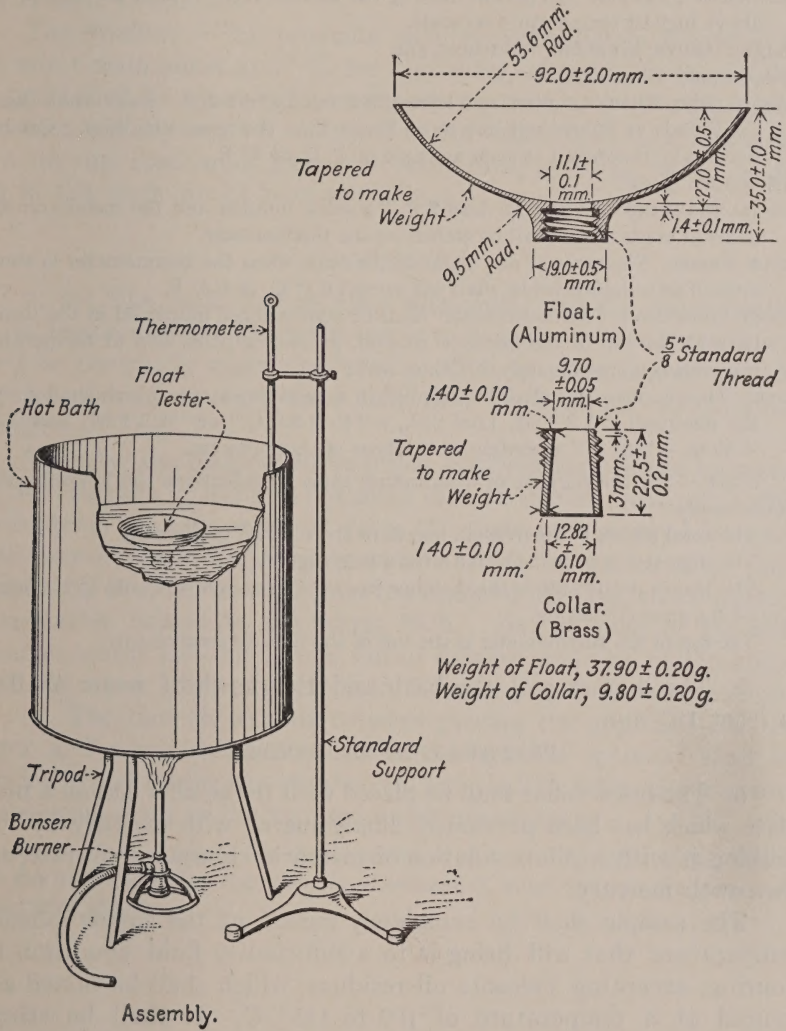


FIG. 1.—Float Test Apparatus.

STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).
BULB: Corning normal or equally suitable thermometric glass.
Length, 9 to 14 mm. (0.35 to 0.55 in.).
Diameter, 4.5 to 5.5 mm. (0.18 to 0.22 in.).

DISTANCE TO 0° C. OR 32° F. LINE FROM BOTTOM OF BULB: 75 to 90 mm. (2.95 to 3.54 in.).

DISTANCE TO 80° C. OR 176° F. LINE FROM TOP OF THERMOMETER: 30 to 45 mm. (1.18 to 1.77 in.).

EXPANSION CHAMBER: To permit heating the thermometer at least 50° C. (90° F.) above highest temperature on scale.

FILLING ABOVE MERCURY: Nitrogen gas.

TOP FINISH: Glass Ring.

GRADUATION: All lines, figures, and letters clear cut and distinct. Each whole degree Centigrade or Fahrenheit line to be longer than the remaining lines. Graduations to be numbered at each multiple of 2° C. or 5° F.

IMMERSION: Total.

SPECIAL MARKING: "A.S.T.M. Low S.P.," a serial number and the manufacturer's name or trade mark shall be etched on the thermometer.

SCALE ERROR: The error at any point of the scale when the thermometer is standardized as provided below shall not exceed 0.2° C. or 0.4° F.

STANDARDIZATION: The thermometer shall be standardized immersed in the testing bath to the top of the mercury column, at the ice point and at temperature intervals of approximately 20° C. or 40° F.

CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. Low S.P., −2 to +80° C." or "A.S.T.M. Low S.P., +30 to +180° F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

Bath.

5. The diameter of the bath and the depth of water shall be at least 185 mm.

PREPARATION OF SAMPLE

Preparation of Sample.

6. The brass collar shall be placed with the smaller end on a brass plate which has been previously amalgamated with mercury by first rubbing it with a dilute solution of mercuric chloride or nitrate, and then with mercury.

The sample shall be completely melted at the lowest possible temperature that will bring it to a sufficiently fluid condition for pouring, excepting creosote oil residues, which shall be mixed and poured at a temperature of 100 to 125° C. It shall be stirred thoroughly until it is homogeneous and free from air bubbles. The sample shall then be poured into the collar in any convenient manner until slightly more than level with the top.

Asphalt and Asphalt Products.—Asphalt and asphalt products shall be cooled to room temperature, placed in water maintained at 5° C. for 5 minutes, after which the surplus material shall be

removed by means of a spatula, or steel knife, which has been slightly heated. The collar and plate shall then be placed in a tin cup containing ice water maintained at 5° C., ±1° C., and left in this bath for at least 15 minutes.

Tar Products.—Tar products shall be immediately immersed in ice water maintained at 5° C. for 5 minutes, after which the surplus material shall be removed by means of a spatula or steel knife, which has been slightly heated. The collar and plate shall then be placed in a tin cup containing ice water maintained at 5° C., ±1° C., and left in this bath for at least 15 minutes. Tar Products.

PROCEDURE

7. (*a*) The bath shall be filled with water and the water heated to the temperature at which the test is to be made. This temperature shall be accurately maintained and shall at no time throughout the test be allowed to vary more than 0.5° C. from the temperature specified. Procedure.

(*b*) After the material to be tested has been kept in the ice water for not less than 15 minutes nor more than 30 minutes, the collar with its contents shall be removed from the plate and screwed into the aluminum float and immersed in water at 5° C. for one minute. Any water shall then be removed from the inside of the float and the latter immediately floated in the warm bath. As the plug of material becomes warm and fluid, it is forced upward and out of the collar until the water gains entrance into the saucer and causes it to sink.

(*c*) The time in seconds between placing the apparatus on the water and when the water breaks through the material shall be determined by means of a stop watch, and shall be taken as a measure of the consistency of the material under examination.

NOTE.—Special precaution should be taken to insure the collar fitting tightly into the float and to see that there is no seepage of water between the collar and float during the test.

STANDARD METHOD OF TEST

FOR

SPECIFIC GRAVITY OF ROAD OILS, ROAD TARS, ASPHALT CEMENTS AND SOFT TAR PITCHES[1]

Serial Designation: D 70 – 27

This method is issued under the fixed designation D 70; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1920; ADOPTED IN AMENDED FORM, 1927.

Specific Gravity.

1. The specific gravity of road oils, road tars, asphalt cements and soft tar pitches shall be expressed as the ratio of the weight of a given volume of the material at 25° C. (77° F.) to that of an equal volume of water at the same temperature and shall be expressed thus:

Specific Gravity, 25° C. (77° F.), ...

Apparatus.

2. The determination of specific gravity shall be made with a pyknometer or weighing bottle (Fig. 1), which shall consist of a straight-walled glass tube approximately 70 mm. long and 22 mm. in diameter, carefully ground to receive an accurately fitting solid glass stopper with a hole of 1.5 to 1.7-mm. bore in place of the usual capillary opening. The lower part of the stopper is made concave in order to allow all air bubbles to escape through the bore. The depth of the cup-shape depression shall be about 4.8 mm. at the center. The stoppered tube should have a capacity of about 24 cc. and when empty should weigh not over 35 g.

Procedure.

3. Before making a determination, the pyknometer with stopper shall first be calibrated by weighing it clean and dry upon an analytical balance. This weight is called "*a*." It shall then be filled with freshly boiled distilled water at a temperature of 25° C. (77° F.), the stopper firmly inserted, all

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

surplus moisture wiped from the surface with a clean dry cloth and again weighed. This weight is called "*b*."

4. When determining the specific gravity of road oils or road tars which flow readily, the material shall be brought to a temperature of 25° C. (77° F.) and poured into the pyknometer until it is full, with care to prevent the inclusion of air bubbles. The stopper is then firmly inserted and all excess of material forced through the opening is carefully removed with a clean dry cloth. The pyknometer and contents are then weighed and this weight is called "*c*." The specific gravity of the material shall be calculated from the formula:

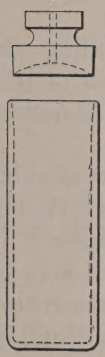


FIG. 1.—Pyknometer or Weighing Bottle.

$$\text{Specific Gravity} = \frac{c-a}{b-a}$$

5. When determining the specific gravity of tar and asphalt products which are too viscous for the method described in Section 4, a small amount of the material shall be brought to a fluid condition by the gentle application of heat, care being exercised to prevent loss by evaporation. When sufficiently fluid, enough is poured into the clean dry pyknometer to about half fill it. Precautions shall be taken to keep the material from touching the sides of the tube above the final level and to prevent the inclusion of air bubbles. The tube should be slightly warmed before filling. The pyknometer and contents are then cooled to room temperature and weighed with the stopper. This weight is called "*c*." The pyknometer is next removed from the balance, filled with freshly boiled distilled water, and the stopper firmly inserted. It is then completely immersed for not less than 30 minutes in a beaker of distilled water maintained at 25° C. (77° F.) after which it is removed, and all surplus water is wiped off with a clean cloth. It is immediately weighed. This weight is called "*d*." The specific gravity of the material shall be calculated from the formula:

$$\text{Specific Gravity} = \frac{c-a}{(b-a)-(d-c)}$$

6. When making the specific gravity determination it is important that: Precautions.

(*a*) Only freshly boiled distilled water shall be used.

(*b*) When weighing the pyknometer completely filled, the temperature of its contents shall be within 1° C. (1.8° F.) of 25° C. (77° F.).

(*c*) Precautions shall be taken to prevent expansion and overflow of the contents from the heat of the hand when wiping the surface of the pyknometer.

(*d*) The presence of all air bubbles shall be eliminated in filling the pyknometer and inserting the stopper.

(*e*) Weighings shall be made quickly after filling the pyknometer and shall be accurate to 1 mg. A number of trial fillings and catch weights may be necessary to obtain the desired degree of accuracy.

(*f*) To prevent breakage of the pyknometer when cleaning it out after a determination has been made upon a very viscous or semi-solid material, it will be found advisable to warm it in an oven at not over 100° C. until most of the material may be poured out and then to swab it with a piece of soft cloth or cotton waste. When cool it may be finally rinsed with carbon disulfide, benzol or other solvent and wiped clean.

Accuracy. 7. The limit of accuracy of the test is ± 0.005 specific gravity.

STANDARD METHOD OF TEST FOR SPECIFIC GRAVITY OF ASPHALTS AND TAR PITCHES SUFFICIENTLY SOLID TO BE HANDLED IN FRAGMENTS[1]

Serial Designation: D 71 – 27

This method is issued under the fixed designation D 71; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1920; ADOPTED IN AMENDED FORM, 1927.

Specific Gravity.

1. The specific gravity of asphalts and tar pitches shall be expressed as the ratio of the weight of a given volume of the material at 25° C. (77° F.) to that of an equal volume of water at the same temperature and shall be expressed thus:

Specific Gravity, 25° C. (77° F.), ...

Apparatus.

2. The determination of specific gravity shall be made with an analytical balance equipped with a pan straddle or other stationary support (Fig. 1).

Test Specimen.

3. The test specimen shall be a cube of the material measuring approximately ½ in. to the edge. It shall be prepared by melting a small sample of the material by the gentle application of heat, care being exercised to prevent loss by evaporation, and pouring when sufficiently fluid into a ½-in. brass cubical mold, which has been amalgamated with mercury and which is placed on an amalgamated brass plate. Precautions should be taken to prevent the inclusion of air bubbles. The hot material should slightly more than fill the mold and when cool the excess may be cut off with a hot spatula. The specimen shall be removed from the mold when cooled to room temperature.

Procedure.

4. The balance shall first be tared with a piece of fine waxed silk thread sufficiently long to reach from the hook on one of the pan supports to the straddle or rest. The test specimen

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

shall then be attached to the thread, so as to be suspended about 1 in. above the straddle from the hook on the pan support, and weighed. This weight is called "*a*" and shall be accurate to 0.1 mg. The specimen, still suspended by the thread, shall then be weighed completely immersed in freshly boiled distilled water at 25° C. (77° F.) ±1° C. (1.8° F.), adhering air bubbles

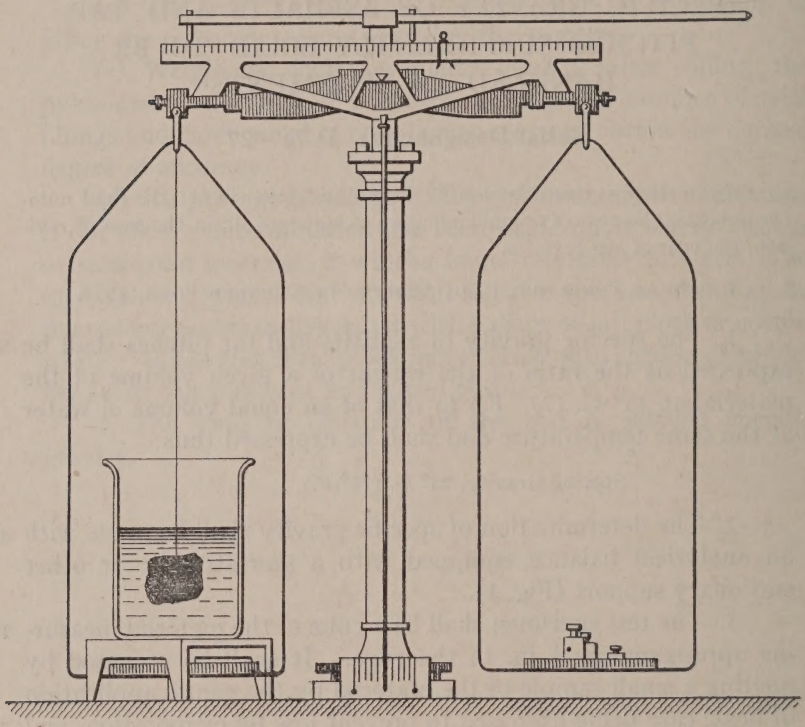


FIG. 1.—Analytical Balance Equipped with Pan Straddle.

being first removed with a fine wire. This weight is called "*b*" and shall also be accurate to 0.1 mg.

The specific gravity of the material shall be calculated from the formula:

$$\text{Specific Gravity} = \frac{a}{a-b}$$

Accuracy. 5. The limit of accuracy of the test is ±0.005 specific gravity.

STANDARD METHODS OF SAMPLING BITUMINOUS MATERIALS[1]

Serial Designation: D 140 – 25

These methods are issued under the fixed designation D 140; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED IN AMENDED FORM, 1925.

1. Samples may be taken for either of two purposes:

General Directions.

(*a*) To represent as nearly as possible an average of the bulk of the material sampled.

(*b*) To ascertain the maximum variation in characteristics which the material may possess. In either case they shall be obtained by methods hereinafter described.

Protection and Preservation.

2. (*a*) Care shall be taken that the samples are not contaminated with dirt or any other extraneous matter and that the sample containers are perfectly clean and dry before filling.

(*b*) Immediately after filling, the sample cantainers shall be tightly closed and properly marked for identification on the container itself or on a linen tag attached to the container.

Time and Place.

3. (*a*) Whenever practical, bituminous materials shall be sampled at the point of manufacture, and at such time as to allow the tests, controlling acceptance or rejection, to be made in advance of shipment.

(*b*) When impracticable to take samples at the point of manufacture, they should be taken from the shipment immediately upon delivery.

Size of Samples.

4. For routine laboratory examination of acceptability of a given lot, not less than one quart of material should be submitted which should be representative of the average sample collected as hereinafter described.

Containers.

5. Containers for liquid bituminous materials shall be small-mouth cans with cork-lined screw caps. Containers for semi-solid and solid materials shall be friction top cans.

SAMPLING AT PLACE OF MANUFACTURE

From Bulk Storage.

6. The inlet and outlet to the storage tank shall be sealed and a 1-gal. sample drawn from the top, middle and bottom contents. The sample may be taken from drain cocks on the side of the tank, if such

[1] Under the standardization procedure of the Society, these methods are under the joint jurisdiction of the A.S.T.M. Committees D-4 on Road and Paving Materials and D-8 on Bituminous Waterproofing and Roofing Materials.

are available. Enough material should be discarded to insure a representative sample. Otherwise, samples may be taken by lowering, weighted bottles or cans into the material. The bottle or can should be fitted with a stopper which can be removed by a string or wire attached to it after it has been lowered to the proper depth. The three samples from bulk storage shall be tested separately for consistency in order to detect stratification. They may then be combined and thoroughly mixed for other tests that may be required.

During Loading.

7. Where tank cars, distributors or barrels are being filled, samples may conveniently be taken from the pipe line through which the material is flowing, as hereinafter described.

(A) When Material is Pumped Under Pressure.

Material Under Pressure.

8. The sampling pipe shall be inserted into a rising section of the pipe line on the discharge side of the pump. The sampling pipe shall be not more than one-eighth the diameter of the line pipe and its opening should be turned at an angle of 90 deg. facing the flow of the liquid. This pipe shall be provided with a plug cock and shall discharge into a receiving drum of 50-gal. capacity. The plug cock shall be so adjusted that there is a steady continuous flow of bituminous material through it and shall be so regulated that the receiver will fill in the same time that is required to make the entire pumping. In the case of semi-solid materials, the receiver shall be provided with a steam coil which shall keep the contents at a temperature just above the liquefying point. At completion of the pumping, the receiver shall be thoroughly agitated, and a 1-qt. sample taken therefrom. The sampling shall be so regulated that for each 1000 gal. of material pumped, at least 1 gal. of sample is taken; but 40 gal. shall be the limit required for any one cargo.

Care should be taken that the drip cock, after once having been set, allows a constant flow during the pumping.

This method is also applicable to gravity flows where the pipe line is completely filled by the outflowing liquid and has a rising section.

(B) When Materials Flow by Gravity.

Material Flowing by Gravity.

9. Materials flowing by gravity through pipes which are not completely filled may be sampled by taking dipperfuls at the outlet at frequent and regular intervals. These samples shall be combined and the total sample shall be not less than 0.1 per cent of the whole material. The samples shall be collected in a receiver and resampled as described in Section 8.

SAMPLING AT POINT OF DELIVERY

10. Samples may be taken by means of a weighted bottle or can, as described in Section 6, or from the unloading pipe line as described in Section 7 Tank Cars.

Liquid bituminous materials shall be sampled before heating. Semi-solid or solid bituminous materials shall be rendered fluid by heating. Sampling should be so conducted as to eliminate the possibility of adventitious water resulting from leaky steam heating coils, rain or snow.

11. Samples may be taken from distributors by means of a weighted bottle or can, as described in Section 6. One sample shall be sufficient. Distributors.

12. (*a*) *Semi-Solid or Solid Materials.*—Where the lot of material to be sampled is obviously from a single run or batch of the producer, one package or cake shall be selected at random and sampled as described in the following paragraph. Where the lot of material to be sampled is not obviously from a single run or batch of the producer, or where the single sample selected as described above fails on test to conform to the requirements of the specifications, a number of packages or cakes shall be selected at random equivalent to the cube root of the total number of packages or cakes in the lot. For convenience, the following table is given, showing the number of samples to be selected for shipments of various sizes: Barrels, Drums and Cakes.

Packages or Cakes in Shipment	Packages or Cakes Selected	Packages or Cakes in Shipment	Packages or Cakes Selected
2 to 8	2	217 to 343	7
9 to 27	3	344 to 512	8
28 to 64	4	513 to 729	9
65 to 125	5	730 to 1000	10
126 to 216	6	1001 to 1331	11

Samples shall be taken from at least 3 in. below the surface and at least 3 in. from the side of the container or cake or from the center of a cake. A clean hatchet may be used if the material is hard enough to shatter and a broad stiff putty knife if the material is soft. An auger, or brace and $\frac{3}{4}$-in. bit, or other suitable means may also be used. When more than one package or cake in a lot is sampled, each individual sample shall be not less than $\frac{1}{4}$ lb. in weight. When the lot of material is obviously from a single run or batch of the producer, all samples from the lot shall be melted and thoroughly mixed, and an average sample taken from the combined material for examination. In case more than a single run or batch of the producer is present and

the batches can be clearly differentiated, a composite sample shall be prepared for examination from each batch. Where it is not possible to differentiate between the various batches each sample shall be examined separately.

(*b*) *Liquid Materials.*—Samples of liquid bituminous materials from barrels or drums shall be secured by taking 1 qt. of material from packages selected at random according to the cube root method described in Paragraph (*a*). When the lot of material is obviously from a single run or batch of the producer the samples shall be combined and thoroughly mixed and an average sample taken from the combined material. In case more than one run or batch is present and can be clearly differentiated, a composite sample shall be prepared from each batch. Where it is impossible to differentiate between the various batches or runs, each sample shall be examined separately.

Bulk and Bags.

13. Solid bituminous materials in crushed fragments or powder, such as gilsonite, grahamite, etc., shall be sampled in accordance with the Standard Method of Sampling Coal (Serial Designation: D 21) of the American Society for Testing Materials.[1]

[1] See p. 530.

STANDARD DEFINITIONS OF TERMS RELATING TO MATERIALS FOR ROADS AND PAVEMENTS[1]

Serial Designation: D 8 – 18

These definitions are issued under the fixed designation D 8; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1912; REVISED, 1915, 1918.

BITUMINOUS MATERIALS

Relating in General to Bituminous Materials:

Bitumens.—Mixtures of native or pyrogenous hydrocarbons and their non-metallic derivatives, which may be gases, liquids, viscous liquids, or solids, and which are soluble in carbon disulfide.

Bituminous.—Containing bitumen or constituting the source of bitumen.

Bituminous Emulsion.—A liquid mixture in which minute globules of bitumen are held in suspension in water or a watery solution.

Cut-Back Products.—Petroleum or tar residuums which have been fluxed with distillates.

Flux.—Bitumens, generally liquid, used in combination with harder bitumens for the purpose of softening the latter.

Liquid Bituminous Materials.—Those having a penetration at 25° C. (77° F.), under a load of 50 g. applied for 1 second, of more than 350.

Semi-Solid Bituminous Materials.—Those having a penetration at 25° C. (77° F.), under a load of 100 g. applied for 5 seconds, of more than 10, and a penetration at 25° C. (77° F.), under a load of 50 g. applied for 1 second, of not more than 350.

Solid Bituminous Materials.—Those having a penetration at 25° C. (77° F.), under a load of 100 g. applied for 5 seconds, of not more than 10.

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

Relating Specifically to Petroleum or Asphalts:

Asphalts.—Solid or semi-solid native bitumens, solid or semi-solid bitumens obtained by refining petroleum, or solid or semi-solid bitumens which are combinations of the bitumens mentioned with petroleums or derivatives thereof, which melt upon the application of heat and which consist of a mixture of hydrocarbons and their derivatives of complex structure, largely cyclic and bridge compounds.

Artificial Asphalt.—Recommended that the use of term be discontinued.

Asphalt Cement.—A fluxed or unfluxed asphalt specially prepared as to quality and consistency for direct use in the manufacture of bituminous pavements, and having a penetration at 25° C. (77° F.) of between 5 and 250, under a load of 100 g. applied for 5 seconds.

Liquid Asphalt.—This is a trade term not subject to definition.

Native Asphalt.—Asphalt occurring as such in nature.

Road Asphalt.—This is a trade term not subject to definition.

Rock Asphalt.—Sandstone or limestone naturally impregnated with asphalt.

Petroleum.—Liquid bitumen occurring as such in nature.

Blown Petroleums.—Semi-solid or solid products produced primarily by the action of air upon liquid native bitumens which are heated during the blowing process.

Topped Petroleum.—Petroleum deprived of its more volatile constituents.

Asphaltenes.—The components of the bitumen in petroleums, petroleum products, malthas, asphalt cements and solid native bitumens, which are soluble in carbon disulphide but insoluble in paraffin naphthas.

Carbenes.—The components of the bitumen in petroleums, petroleum products, malthas, asphalt cements and solid native bitumens, which are soluble in carbon disulphide but insoluble in carbon tetrachloride.

Relating Specifically to Tars and Pitches:

Tars.—Bitumens which yield pitches upon fractional distillation and which are produced as distillates by the destructive distillation of bitumens, pyrobitumens or organic materials.

Coal Tar.—The mixture of hydrocarbon distillates, mostly unsaturated ring compounds, produced in the destructive distillation of coal.

Gas-House Coal Tar.—Coal tar produced in gas-house retorts in the manufacture of illuminating gas from bituminous coal.

Coke-Oven Tar.—Coal tar produced in by-product coke ovens in the manufacture of coke from bituminous coal.

Oil-Gas Tars.—Tars produced by cracking oil vapors at high temperatures in the manufacture of oil gas.

Water-Gas Tars.—Tars produced by cracking oil vapors at high temperatures in the manufacture of carburetted water-gas.

Dehydrated Tars.—Tars from which all water has been removed.

Refined Tar.—Tar freed from water by evaporation or distillation which is continued until the residue is of desired consistency; or a product produced by fluxing tar residuum with tar distillate.

Dead Oils.—Oils with a density greater than water which are distilled from tars.

Pitches.—Solid residues produced in the evaporation or distillation of bitumens, the term being usually applied to residues obtained from tars.

Straight-run Pitch.—A pitch run to the consistency desired, in the initial process of distillation, without subsequent fluxing.

Free Carbon in Tars.—Organic matter which is insoluble in carbon disulfide.

Relating Specifically to Tests:

Normal Temperature.—As applied to laboratory observations of the physical characteristics of bituminous materials, is 25° C. (77° F.).

Consistency.—The degree of solidity or fluidity of bituminous materials.

Fixed Carbon.—The organic matter of the residual coke obtained upon burning hydrocarbon products in a covered vessel in the absence of free oxygen.

Penetration.—The consistency of a bituminous material expressed as the distance that a standard needle vertically penetrates a sample of the material under known conditions of loading, time and temperature. Where the conditions of test are not specifically mentioned, the load, time and temperature are understood to be 100 g., 5 seconds, and 25° C. (77° F.),

respectively, and the units of penetration to indicate hundredths of a centimeter.

Viscosity.—The measure of the resistance to flow of a bituminous material, usually stated as the time of flow of a given amount of the material through a given orifice.

NON-BITUMINOUS MATERIALS

Relating in General to Non-Bituminous Materials:

Aggregate.—The inert material, such as sand, gravel, shell, slag or broken stone or combinations thereof, with which the cementing material is mixed to form a mortar or concrete.

Matrix.—The binding material or mixture of binding material and fine aggregate in which the large aggregate is embedded or held in place.

Crusher-Run.—The total unscreened product of a stone crusher.

Stone Chips.—Small angular fragments of stone containing no dust.

Tailings.—Stones which after going through the crusher do not pass through the largest openings of the screen.

Rubble.—Rough stones of irregular shapes and sizes, broken from larger masses either naturally or artificially, as by geological action, in quarrying, or in stone cutting or blasting.

Relating Specifically to Materials:

Bank Gravel.—Gravel found in natural deposits, usually more or less intermixed with fine material, such as sand or clay, or combinations thereof; gravelly clay, gravelly sand, clayey gravel and sandy gravel, indicate the varying proportions of the materials in the mixture.

Clinker.—Generally a fused or partly fused by-product of the combustion of coal, but also including lava and Portland-cement clinker, and partly vitrified slag and brick.

Chert.—Compact siliceous rock formed of calcedonic or opaline silica, or both.

Granite.—A granitoid igneous rock consisting of quartz, orthoclase, more or less oligoclase, biotite and muscovite.

Granitoid.—A textural term to describe those igneous rocks which are entirely composed of recognizable minerals.

Slag.—Fused or partly fused compounds of silica in combination with lime or other bases, resulting in secondary products from the reduction of metallic ores.

Soil.—A mixture of fine earthy material with more or less organic matter resulting from the growth and decomposition of vegetation or animal matter.

Relating Specifically to Tests:

Mesh.—The square opening of a sieve.

Screen.—In laboratory work an apparatus, in which the apertures are circular, for separating sizes of material.

Sieve.—In laboratory work an apparatus, in which the apertures are square, for separating sizes of material.

STANDARD SPECIFICATIONS
FOR
FOUNDRY COKE[1]

Serial Designation: D 17–16

These specifications are issued under the fixed designation D 17; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1916.

CHEMICAL PROPERTIES AND TESTS

(*A*) *Sampling.*

Unit of Sampling.

1. Each carload, or its equivalent, shall be considered as a unit.

Method of Sampling.

2. (*a*) The sample shall be taken from the exposed surface of the car, by knocking off with a hammer a piece approximately the size of a walnut, at regular intervals of 18 in. along three lines running from one end of the car to the other. One of these lines shall pass through the center of the car and the other two lines shall be 2 ft. from the respective sides of the car.

(*b*) The intervals of sampling along the three lines may be measured by using a hammer with a handle 18 in. long, breaking off a piece of coke the size of a walnut at each point where the head of the hammer rests, regardless of the appearance of the particular piece that happens to occur under the head of the hammer.

Quantity of Gross Sample.

3. The total quantity of sample collected in the above manner shall not be less than 1 cu. ft.

Reduction of Gross Sample, Without Regard to Moisture.

4. When the total moisture content is not to be determined, the entire gross sample shall be crushed to pass through a 4760-micron (No. 4) sieve, under such conditions as shall prevent loss or the accidental admixture of foreign matter. The crushing shall be done mechanically with a jaw crusher, or by hand on a chilled iron or hard steel plate by impact of a chilled iron or hard steel tamping bar, hammer or sledge, avoiding all rubbing action, otherwise the ash content of the sample

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-5 on Coal and Coke.

will be materially increased by the addition of iron from the crushing apparatus, even though hardened steel or chilled iron is used.

After all the gross sample has been passed through the 4760-micron (No. 4) sieve, it shall be mixed on a strong, closely woven cloth about 5 ft. square by raising successively the four sides of the cloth, thus rolling the sample about until thoroughly mixed. The four corners of the cloth shall then be gathered up, and the sample shall be formed in a conical pile and reduced in quantity by quartering as follows:

The cone shall be flattened, its apex being pressed down so that each quarter contains the material originally in it. The flattened mass shall then be divided into four equal quarters. The diagonally opposite quarters shall then be removed and discarded and the space that they occupied brushed clean. The two remaining quarters shall be successively mixed, coned and quartered on the cloth as before, until two opposite quarters shall weigh not less than 5 lb., which shall then be placed in a suitable container for transportation to the laboratory. In case duplicate laboratory samples are desired, the rejected portions of the original sample passing the 4760-micron (No. 4) sieve shall be combined, mixed and quartered down to a similar 5-lb. sample.

Optional Use of Standard Sample for Moisture Determination.

5. The sample prepared by the above method may, at the option of the purchaser, be used for an approximate moisture determination. In such cases the gross sample shall be immediately crushed and reduced to the 5-lb. laboratory sample as rapidly as possible, to minimize the loss of moisture. The container for shipment to the laboratory shall be moisture-tight. Since the sample obtained by this method will usually show less than the true moisture content of the gross sample, the purchaser shall have the privilege of a special moisture sample as hereinafter provided, if the standard sample shows more than 3 per cent moisture.

Special Moisture Sample.

6. The special moisture sample shall consist of not less than 1 cu. ft. of walnut size. It shall be taken in the manner described in Section 2, and shall be placed, immediately after collection, in a moisture-tight container for transportation to the laboratory. The car shall be weighed at the time the special moisture sample is collected.

Resampling.

7. In case of disagreement between buyer and seller, an independent chemist, mutually agreed upon, shall be employed to sample and analyze the coke, the cost to be borne by the party at fault.

The resample shall be taken and prepared as prescribed in the foregoing sections, except that the minimum quantity of gross sample shall be not less than 1 bushel in volume, taken at intervals of 18 in. on six equidistant lines parallel to the side of the car.

(*B*) *Chemical Analysis.*

Method of Analysis.

8. The sample received at the laboratory shall be prepared for analysis, and the percentage of moisture, volatile matter, fixed carbon, ash, and sulfur shall be determined as specified in the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (Serial Designation: D 271) of the American Society for Testing Materials.[1]

Chemical Composition.

9. The dry coke shall not exceed the following limits in chemical composition:

Volatile matter	not over	2.0 per cent
Fixed carbon	not under	86.0 "
Ash	not over	12.0 "
Sulfur	" "	1.0 "

REJECTION

Deduction for Excess Moisture.

10. (*a*) In case the original standard sample was taken in accordance with Section 5 and showed more than 3 per cent moisture, the purchaser shall have the option of taking a special moisture sample according to Section 6, and of deducting the moisture found in excess of 3 per cent, from the weight of coke found on reweighing the car at the time the special moisture sample was taken.

(*b*) In case the original standard sample was taken with special regard to moisture in accordance with Section 6, the purchaser shall have the option of deducting the moisture in excess of 3 per cent from the weight of coke, provided that the car was weighed at the time of sampling.

Rejection.

11. Coke which fails to conform to the limits of chemical composition given in Section 9 will be rejected, and the seller shall be notified within 5 working days from the date of sampling.

[1] See p. 535.

STANDARD SPECIFICATIONS
FOR
GAS AND COKING COALS[1]

Serial Designation: D 166 – 24

These specifications are issued under the fixed designation D 166; the fina number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED, 1924.

Scope. 1. Gas and coking coals must yield both merchantable gas and coke when distilled in a retort or oven by commercial methods. The type of coals may vary within rather wide limits according to the treatment in the retort and the market for the products. These specifications, therefore, merely give the limits within which gas and coking coals will usually fall, and indicate the circumstances under which further restrictive conditions should be imposed.

SAMPLING AND ANALYSIS

Sampling. 2. The coal shall be sampled in accordance with the Standard Method of Sampling Coal (Serial Designation: D 21) of the American Society for Testing Materials.[2]

Analysis. 3. Analyses of the coal, when required, shall be made in accordance with the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (Serial Designation: D 271) of the American Society for Testing Materials.[3]

CHEMICAL AND PHYSICAL PROPERTIES

The carbon ratio, that is, the ratio of fixed carbon to volatile matter, while not entirely reliable, is the best simple index to the behavior of the coal when carbonized. The carbon ratio in the case of gas coals will vary from 1.4 to 2.0 and for coking coals from 1.4 to 5.0. The latter includes a wide range of coals varying from high volatile gas coal to low volatile or "smokeless" coal.

Mine Moisture. 4. (*a*) The percentage of moisture in the coal as mined shall be subject to agreement by the purchaser and the seller.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-5 on Coal and Coke.

These specifications agree with those of the American Gas Association and were prepared jointly by that organization and the American Society for Testing Materials.

[2] See p. 530. [3] See p. 535.

(*b*) In the absence of a definite agreement between the purchaser and the seller, the mine moisture in the coal as mined shall not exceed 4.0 per cent. The moisture shall be determined by the general average composition of coal from the mine in question and an analysis of each shipment shall not be required.

Fusion Point of Ash.

5. The fusion temperature of ash of coal, the coke from which is intended for domestic and industrial use, shall not be below 2200° F. In the case of coke for use in the manufacture of water gas, the fusion temperature of the ash of the coal shall preferably be higher than 2300° F. The fusion temperature of the ash shall be determined in accordance with the method for determination of fusibility of coal ash appearing in the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (Serial Designation: D 271) of the American Society for Testing Materials.[1]

NOTE.—The fusion point of ash is not usually important for metallurgical work. It is important, however, in the case of coke for domestic and industrial furnace use and for the manufacture of water gas.

A. Special Requirements for Gas Coals

Volatile Matter.

6. Gas coal shall contain not less than 35.0 per cent of volatile matter when determined on the moisture and ash-free basis.

NOTE.—This is equivalent to 30.8 per cent volatile matter for a coal containing 12.0 per cent of combined ash and moisture.

Ash.

7. In the case of gas coals, the ash in the dry coal shall be not over 9 per cent.

Sulfur.

8. The composition of gas coal shall be such that the dry coke produced therefrom will contain not over 1.5 per cent of sulfur and the resultant gas will contain not more than 30 grains of sulfur, in the form of compounds other than hydrogen sulfide, per 100 cu. ft. of gas.

Quality of Coke.

9. Gas coal shall be such that the coke produced therefrom will be of sufficient size and strength to be suitable for domestic use or for the manufacture of water gas.

NOTE.—These physical characteristics of coke are not amenable to simple explicit definition and must necessarily be left to the judgment of experienced operators.

B. Special Requirements for Coking Coals

Ash.

10. In the case of coking coals, the ash in the dry coal shall be not over 9 per cent.

Sulfur.

11. (*a*) If metallurgical coke is to be produced, the composition of the coking coal shall be such that the dry coke produced therefrom

[1] See p. 535.

will not contain more than 1.0 per cent of sulfur in the case of foundry coke, and 1.3 per cent of sulfur in the case of blast-furnace coke.

(*b*) If gas is to be sold for dometic use, the composition of the coking coal shall be such that the resultant gas will contain not more than 30 grains of sulfur, in the form of compounds other than hydrogen sulfide, per 100 cu. ft. of gas.

12. A limitation as to phosphorus, which may be required when coke is used for metallurgical purposes, shall be subject to agreement between the purchaser and the seller. Phosphorus.

13. The composition of coking coal which is to be charged into a by-product oven without admixture shall be such that the coke produced therefrom will shrink sufficiently to permit of its being discharged from the oven without difficulty. Shrinkage.

NOTE.—The mixing of coals for by-product coke-oven use is widely practiced, and such mixtures usually contain, as a very important component, low volatile or "smokeless" coking coals, which when carbonized alone would not give the requisite shrinkage.

14. The composition of the coking coal shall be such that the coke produced therefrom will meet such requirements as to size, strength, and structure as are necessary for good practice in the industries using the coke. Quality of Coke.

NOTE.—These physical characteristics of coke are not amenable to simple explicit definition and must necessarily be left to the judgment of experienced operators.

STANDARD METHOD
OF
SAMPLING COAL[1]

Serial Designation: D 21 – 16

This method is issued under the fixed designation D 21; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1916.

This method was approved March 28, 1921, as "Tentative American Standard" by the American Engineering Standards Committee.

It is imperative that every sample be collected and prepared carefully and conscientiously and in strict accordance with the standard methods described herein, for if the sampling is improperly done, the sample will be in error, and it may be impossible or impracticable to take another sample; but if an analysis is in error, another analysis can easily be made of the original sample.

Gross samples of the quantities designated herein must be taken whether the coal to be sampled consists of a few tons or several hundred tons, because of the following cardinal principle in sampling coal that must be recognized and understood; that is, the effect of the chance inclusion or exclusion of too many or too few pieces of slate or other impurities in what, or from what, would otherwise have been a representative sample will cause the analysis to be in error accordingly, regardless of the tonnage sampled. For example, the chance inclusion or exclusion of 10 lb. too much or too little of impurities in or from an otherwise representative sample of 100 lb. would cause the analysis to show an error in ash content and in heat units of approximately 10 per cent, whereas for a 1000-lb. sample, the effect would be approximately only 1 per cent, being the same whether the sample is collected from a 1-ton lot or from a lot consisting of several hundred tons.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-5 on Coal and Coke.

When this method of sampling is to be employed as a part of any contract or agreement, the following provisions shall be specifically agreed to by the parties to such contract or agreement:

(*a*) The place at which the coal is to be sampled (see Section 1);

(*b*) The approximate size of the sample required when the standard conditions do not apply (see Section 3);

(*c*) The number of samples to be taken or the amount of coal to be represented by each sample when the standard conditions do not apply (see Section 4).

FOR ALL DETERMINATIONS EXCEPT TOTAL MOISTURE

Time of Sampling.

1. The coal shall be sampled when it is being loaded into or unloaded from railroad cars, ships, barges, or wagons, or when discharged from supply bins, or from industrial railway cars, or grab buckets, or from any coal-conveying equipment, as the case may be. If the coal is crushed as received, samples usually can be taken advantageously after the coal has passed through the crusher. Samples collected from the surface of coal in piles or bins, or in cars, ships or barges are generally unreliable.

Size of Increments.

2. To collect samples, a shovel or specially designed tool, or mechanical means shall be used for taking equal portions or increments. For slack or small sizes of anthracite, increments as small as 5 to 10 lb. may be taken, but for run-of-mine or lump coal, increments should be at least 10 to 30 lb.

Collection of Gross Sample.

3. The increments shall be regularly and systematically collected, so that the entire quantity of coal sampled will be represented proportionately in the gross sample, and with such frequency that a gross sample of the required amount shall be collected. The standard gross sample shall not be less than 1000 lb., except that for slack coal and small sizes of anthracite in which the impurities do not exist in abnormal quantities or in pieces larger than $\frac{3}{4}$ in., a gross sample of approximately 500 lb. shall be considered sufficient. If the coal contains an unusual amount of impurities, such as slate, and if the pieces of such impurities are very large, a gross sample of 1500 lb. or more shall be collected. The gross sample should contain the same proportion of lump coal, fine coal, and impurities as is contained in the coal sampled. When coal is extremely lumpy, it is best to break a proportional amount of the lumps before taking the various increments of a sample. Provision should be made for the preservation of the integrity of the sample.

Quantity Represented.

4. A gross sample shall be taken for each 500 tons or less, or in case of larger tonnages, for such quantities as may be agreed upon.

Crushing.

5. After the gross sample has been collected, it shall be systematically crushed, mixed, and reduced in quantity to convenient size for transmittal to the laboratory. The sample may be crushed by hand or by any mechanical means, but under such conditions as shall prevent loss or the accidental admixture of foreign matter. Samples of the quantities indicated in Table I shall be crushed so that no pieces of coal and impurities will be greater in any dimension, as judged by eye, than specified for the sample before division into two approximately equal parts.

The method of reducing by hand the quantity of coal in a gross sample shall be carried out as prescribed in Section 6,

TABLE I.

Weight of Sample to be Divided, lb.	Largest Size of Coal and Impurities Allowable in Sample before Division, in.
1000 or over	1
500	$\frac{3}{4}$
250	$\frac{1}{2}$
125	$\frac{3}{8}$
60	$\frac{1}{4}$
30	$\frac{3}{16}$ or to pass a 4760-micron (No. 4) sieve

even should the initial size of coal and impurities be less than indicated in Table I.

Hand Preparation.

6. The progressive reduction in the weight of the sample to the quantities indicated in Table I shall be done by the following methods, which are illustrated in Plate V:

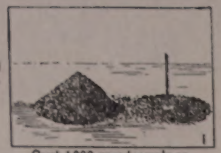
Mixing and Reduction by Discarding Alternate Shovelsful.

(*a*) The alternate-shovel method of reducing the gross sample shall be repeated until the sample is reduced to approximately 250 lb., and care shall be observed before each reduction in quantity that the sample has been crushed to the fineness prescribed in Table I.

The crushed coal shall be shoveled into a conical pile (Figs. 2 or 7, Plate V) by depositing each shovelful of coal on top of the preceding one, and then formed into a long pile in the following manner: The sampler shall take a shovelful of coal from the conical pile and spread it out in a straight line (Figs. 3*A* or 8*A*) having a width equal to the width of the shovel and a length of 5 to 10 ft. His next shovelful shall be spread directly over the

STANDARD METHOD OF SAMPLING COAL.

FIRST STAGE IN THE PREPARATION OF A 1,000-POUND SAMPLE

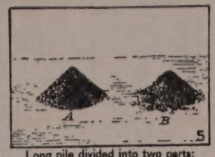


1. Crush 1,000-pound sample on hard, clean surface to 1" size

2. 1,000-pound sample crushed to 1" and coned

3. Mix by forming long pile. *A*—spreading out first shovelful. *B*—long pile completed

4. Halving by alternate shovel method. Shovelfuls 1, 3, 5, etc., reserved as 5, *A*; 2, 4, 6, etc., rejected as 5, *B*

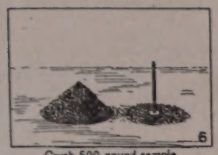


5. Long pile divided into two parts; *A*—reserve; *B*—reject

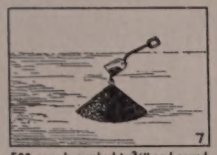
NOTE

SELECT A HARD, CLEAN SURFACE, FREE OF CRACKS AND PROTECTED FROM RAIN, SNOW, WIND, AND BEATING SUN. DO NOT LET CINDERS, SAND, CHIPPINGS FROM FLOOR, OR ANY OTHER FOREIGN MATTER GET INTO THE SAMPLE. PROTECT SAMPLE FROM LOSS OR GAIN IN MOISTURE

SECOND STAGE



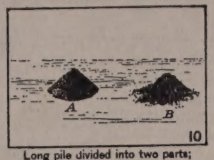
6. Crush 500-pound sample (fig. 5, *A*) to ¾" size



7. 500 pounds crushed to ¾" and coned

8. Mix by forming long pile. *A*—spreading out first shovelful. *B*—long pile completed

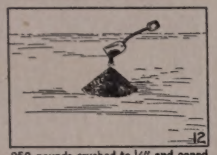
9. Halving by alternate shovel method. Shovelfuls 1, 3, 5, etc., reserved as 10, *A*; 2, 4, 6, etc., rejected as 10, *B*



10. Long pile divided into two parts; *A*—reserve; *B*—reject

THIRD STAGE

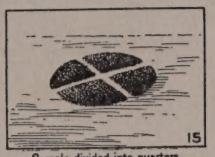
11. Crush 250-pound sample (fig. 10, *A*) to ½" size



12. 250-pounds crushed to ½" and coned

13. Mix by forming new cone

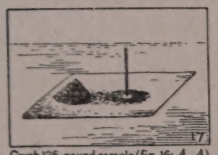
14. Quarter after flattening cone



15. Sample divided into quarters

16. Retain opposite quarters *A*, *A*. Reject quarters *B*, *B*

FOURTH STAGE

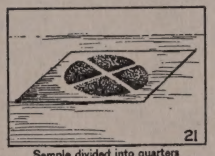


17. Crush 125-pound sample (fig. 16: *A*, *A*) on blanket to 3/16" size

18. Mix by rolling on blanket

19. Form cone after mixing

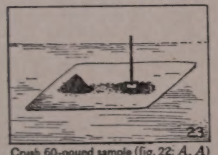
20. Quarter after flattening cone



21. Sample divided into quarters

22. Retain opposite quarters *A*, *A*. Reject quarters *B*, *B*

FIFTH STAGE

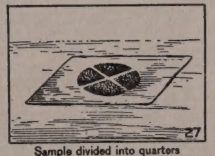


23. Crush 60-pound sample (fig. 22: *A*, *A*) to ¼" size

24. Mix by rolling on blanket

25. Form cone after mixing

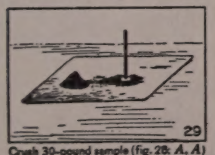
26. Quarter after flattening cone



27. Sample divided into quarters

28. Retain opposite quarters *A*, *A*. Reject quarters *B*, *B*

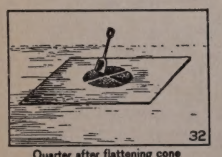
SIXTH STAGE



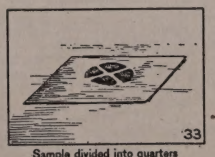
29. Crush 30-pound sample (fig. 28: *A*, *A*) to ⅛" or to pass a 4760-micron (No. 4) Sieve

30. Mix by rolling on blanket

31. Form cone after mixing



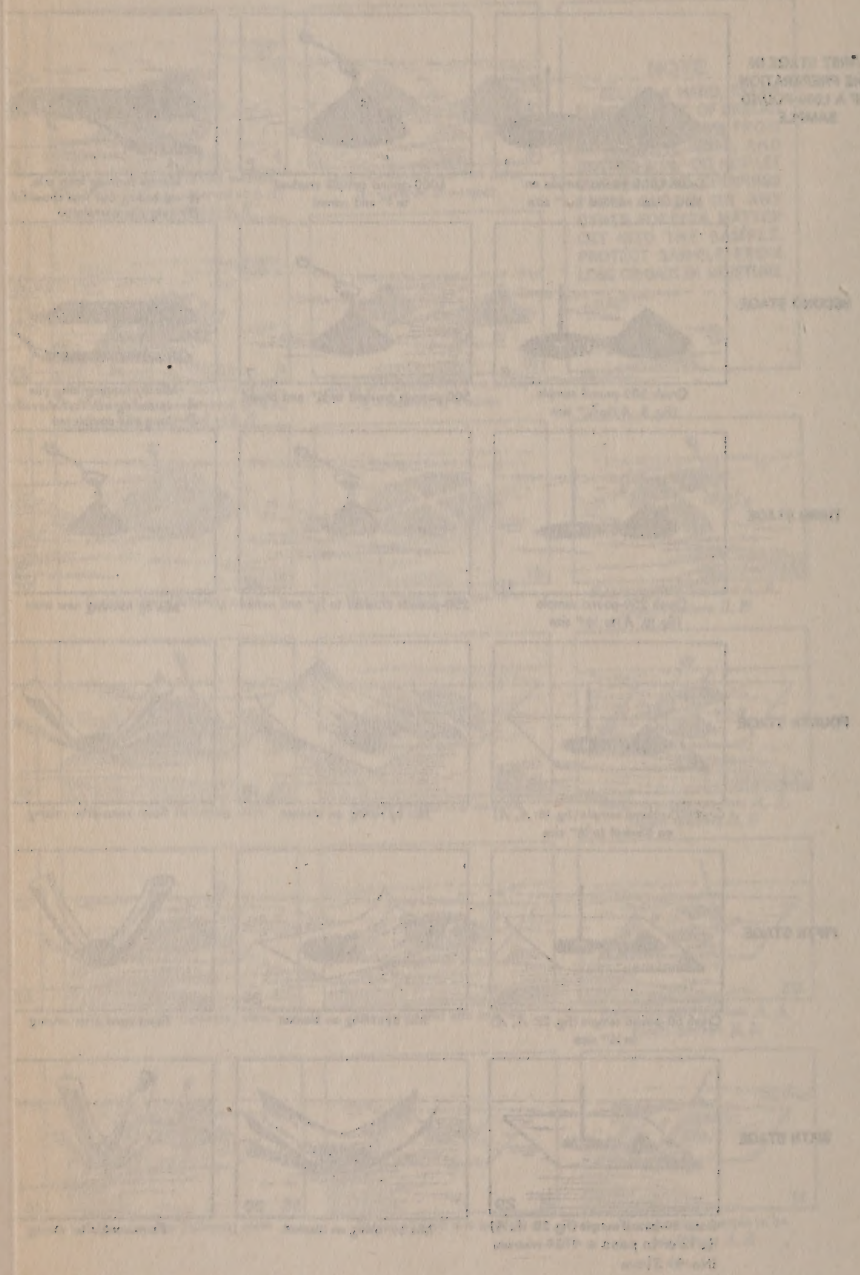
32. Quarter after flattening cone



33. Sample divided into quarters

34. The laboratory sample to be taken from *A*, *A*

NECESSARY TOOLS: SHOVEL, TAMPER, BLANKET (MEASURING ABOUT 6 BY 8 FT.), BROOM, AND RAKE. USE RAKE FOR RAKING OVER COAL WHEN CRUSHING IT, SO THAT ALL LUMPS WILL BE CRUSHED. SWEEP FLOOR OR BLANKET CLEAN OF ALL DISCARDED COAL AFTER EACH TIME SAMPLE IS HALVED OR QUARTERED.



top of the first shovelful, but in the opposite direction, and so on back and forth, the pile being occasionally flattened, until all the coal has been formed into one long pile (Figs. 3*B* or 8*B*). The sampler shall then discard half of this pile, proceeding as follows:

Beginning on one side of the pile, at either end, and shoveling from the bottom of the pile, the sampler shall take one shovelful (shovelful No. 1, Figs. 4 or 9) and set it aside; advancing along the side of the pile a distance equal to the width of the shovel, he shall take a second shovelful (shovelful No. 2) and discard it; again advancing in the same direction one shovel width, he shall take a third shovelful (shovelful No. 3) and add it to the first. The fourth (shovelful No. 4) shall be taken in a like manner and discarded, the fifth (shovelful No. 5) retained, and so on, the sampler advancing always in the same direction around the pile so that its size will be gradually reduced in a uniform manner. When the pile is removed, about half of the original quantity of coal should be contained in the new pile formed by the alternate shovelsful which have been retained. (Figs. 5*A* or 10*A* show the retained halves, and Figs. 5*B* or 10*B* the rejected halves.)

Mixing and Reduction by Quartering.

(*b*) After the gross sample has been reduced by the above method to approximately 250 lb., further reduction in quantity shall be by the quartering method. Before each quartering, the sample shall be crushed to the fineness prescribed in Table I.

Quantities of 125 to 250 lb. shall be thoroughly mixed by coning and re-coning (Figs. 12 and 13); quantities less than 125 lb. shall be placed on a suitable cloth, measuring about 6 by 8 ft., mixed by raising first one end of the cloth and then the other (Figs. 18, 24 or 30), so as to roll the coal back and forth, and after being thoroughly mixed shall be formed into a conical pile by gathering together the four corners of the cloth (Figs. 19, 25 or 31). The quartering of the conical pile shall be done as follows:

The cone shall be flattened, its apex being pressed vertically down with a shovel, or board, so that after the pile has been quartered, each quarter will contain the material originally in it. The flattened mass, which shall be of uniform thickness and diameter, shall then be marked into quarters (Figs. 14, 20, 26 or 32) by two lines that intersect at right angles directly under a point corresponding to the apex of the original cone. The diagonally opposite quarters (*BB* in Figs. 16, 22, 28 or 34)

shall then be shoveled away and discarded and the space that they occupied brushed clean. The coal remaining shall be successively crushed, mixed, coned, and quartered until the sample is reduced to the desired quantity.

(*c*) The 30-lb. quantity (Fig. 29) shall be crushed to $\frac{3}{16}$-in. size or to pass a 4760-micron (No. 4) sieve, mixed, coned, flattened and quartered. The laboratory samples shall include all of one of the quarters, or all of two opposite quarters (Fig. 34), as may be required. The laboratory sample shall be immediately placed in a suitable container and sealed in such a manner as to preclude tampering.

Mechanical Preparation.

7. Only such mechanical means as will give equally representative samples shall be used in substitution for the hand method of preparation herein standardized.

FOR THE DETERMINATION OF TOTAL MOISTURE

Collection of Moisture Sample.

8. The special moisture sample shall weigh approximately 100 lb., and shall be accumulated by placing in a waterproof receptacle with a tight-fitting and waterproof lid small equal parts of freshly taken increments of the standard gross sample. The accumulated moisture sample shall be rapidly crushed and reduced mechanically or by hand to about a 5-lb. quantity, which shall be immediately placed in a container and sealed air-tight and forwarded to the laboratory without delay.

Use of Standard Sample.

9. Only when equally representative results will be obtained shall the standard gross sample be used instead of the special moisture sample for the determination of total moisture.

STANDARD METHODS OF LABORATORY SAMPLING AND ANALYSIS OF COAL AND COKE[1]

Serial Designation: D 271 – 27

These methods are issued under the fixed designation D 271; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1916, 1918; REVISED, 1921, 1924, 1927.

PREPARATION OF LABORATORY SAMPLES

APPARATUS

(*A*) *For Coal:*

1. The apparatus used for sampling coal shall consist of the following: **Apparatus for Sampling Coal.**

(*a*) *Air Drying Oven.*—An oven for air-drying wet samples. A suggested form is shown in Fig. 1. Such an oven is not essential but is economical where many samples are to be dried.[2]

(*b*) *Pans for Air-Drying Wet Samples.*—Galvanized iron pans 18 by 18 in. by 1.5 in. in depth.

(*c*) *Balance or Solution Scale.*—A balance or scale having a capacity of 5 kg. sensitive to 0.5 g. for weighing the galvanized iron pans with samples.

(*d*) *Crusher.*—A jaw crusher suitable for crushing coarse samples to pass a 4760-micron (No. 4) sieve.

(*e*) *Grinder.*—A roll crusher or coffee-mill type of grinder suitable for reducing the material passing a 4760-micron (No. 4) sieve to pass an 840-micron (No. 20) sieve. To reduce the moisture loss while crushing, a coffee-mill type of grinder should be entirely enclosed and have an enclosed hopper and receptacle capable of holding 10 lb. of coal.

(*f*) *Pulverizer.*—An Abbé ball mill, planetary disk crusher, chrome-steel bucking board, or any satisfactory form of pulverizer

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-5 on Coal and Coke. They are a combination of the previous Standard Methods of Laboratory Sampling and Analysis of Coal and of Laboratory Sampling and Analysis of Coke, last published under the serial designations D 22 – 24 and D 37 – 24, respectively.

These methods agree with those of the American Chemical Society and were prepared jointly by that organization and the American Society for Testing Materials.

[2] For details of air-drying oven, see F. M. Stanton and A. C. Fieldner, "Methods of Analyzing Coal and Coke," *Technical Paper No. 8*, Bureau of Mines, p. 2 (1926).

for reducing the material passing an 840-micron (No. 20) sieve to pass a 250-micron (No. 60) sieve. The porcelain jars for the ball mill should be approximately 9 in. in diameter and 10 in. high. The flint pebbles should be smooth, hard and well rounded.

(*g*) *Large Riffle Sampler.*—A large riffle sampler with $\frac{1}{2}$ or $\frac{5}{8}$-in. divisions for reducing the coal passing the 4760-micron (No. 4) sieve to 10 lb. (see Fig. 2 (*a*)).

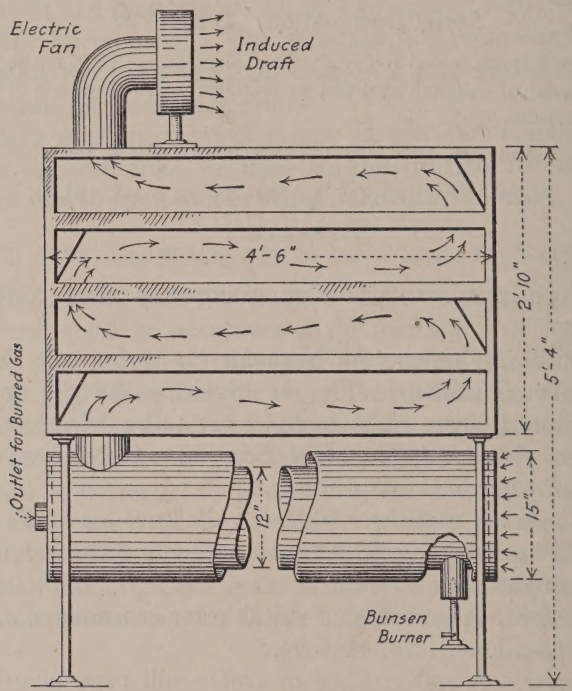


FIG. 1.—Drier for Coarse Samples. The outlet for air at the top may be connected with a chimney or any other device which will furnish a suitable draft. (*Bulletin No. 9*, Geological Survey of Ohio, p. 312.)

(*h*) *Small Riffle Sampler.*—A small riffle sampler with $\frac{1}{4}$ or $\frac{3}{8}$-in. divisions for reducing the material passing the 840-micron (No. 20) and the 250-micron (No. 60) sieves to a laboratory sample (see Fig. 2 (*b*)).

(*i*) *Sieve.*—An 8-in. 250-micron (No. 60) sieve with cover and receiver.

NOTE.—The sieve designations employed are those of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials,[1] and all sieves shall conform to the detailed requirements of these specifications.

[1] See p. 917.

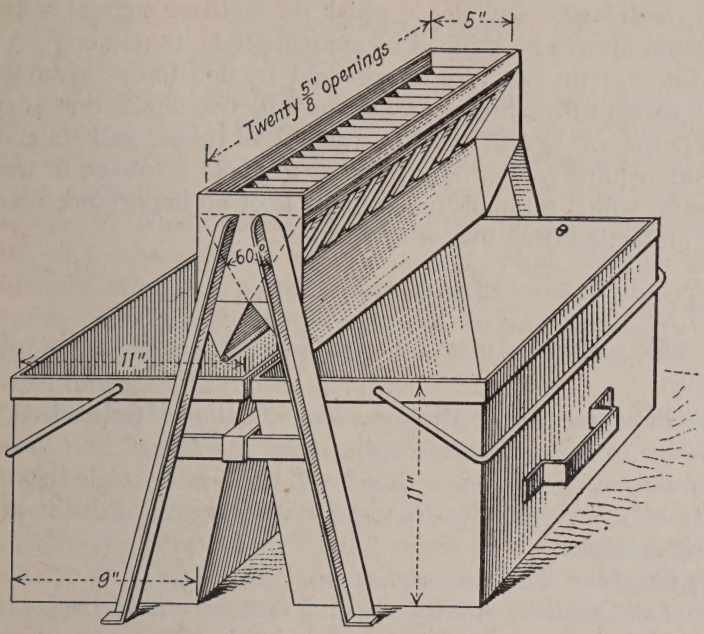


FIG. 2 (*a*).—Large Riffle Sampler.

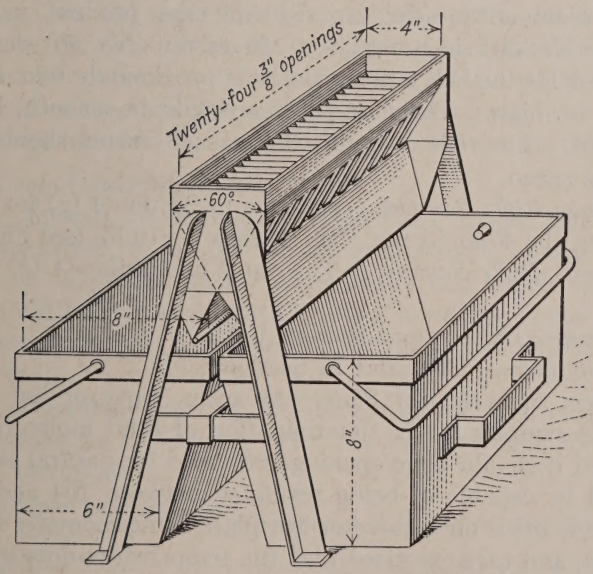


FIG. 2 (*b*).—Small Riffle Sampler.

(*j*) *Containers.*—Samples in which the moisture content is important should always be shipped in moisture-tight containers. A galvanized-iron or tin can with an air-tight friction top or a screw top which is sealed with a rubber gasket and adhesive tape is best adapted to this purpose. Glass fruit jars sealed with rubber gaskets may be used, but require very careful packing to avoid breakage in transit. Samples in which the moisture content is of no importance need no special protection from loss of moisture.

(*B*) *For Coke:*

Apparatus for Sampling Coke.

2. The apparatus used for sampling coke shall consist of the following:

(*a*) *Pans for Total Moisture Determination.*—Galvanized iron pans 24 by 24 in. by 4 in. in depth.

(*b*) *Balance or Solution Scale.*—A balance or scale having a capacity of 10 kg. sensitive to 1 g. for weighing the galvanized-iron pans with samples.

(*c*) *Crusher.*—The same as Section 1 (*d*).

(*d*) *Roll-Crusher.*—A hard-steel roll crusher suitable for reducing the material passing a 4760-micron (No. 4) sieve to pass an 840-micron (No. 20) sieve.

(*e*) *Pulverizer.*—An Abbé ball mill, hard-steel roll crusher or hard-steel diamond mortar for reducing the product passing an 840-micron (No. 20) sieve to pass a 250-micron (No. 60) sieve. The porcelain jars for the ball mill should be approximately 9 in. in diameter and 10 in. high. The flint pebbles should be smooth, hard and well rounded. The rolls of the hard-steel roll crusher should revolve at the same speed.

(*f*) *Large Riffle Sampler.*—Same as in Section 1 (*g*) for reducing coke passing the 4760-micron (No. 4) sieve to 10 lb. (see Fig. 2 (*a*)).

(*g*) *Small Riffle Sampler.*—The same as Section 1 (*h*) (see Fig. 2 (*b*)).

(*h*) *Sieve.*—The same as Section 1 (*i*).

(*i*) *Containers.*—The same as Section 1 (*j*).

(*j*) *Oven, Stove or Hot Plate.*—An oven, stove or hot plate for drying coke samples in the determination of total moisture. If an oven is used it should have openings provided for natural ventilation and should be capable of being regulated between 104 and 200° C. If the coke is dried on a stove or hot plate a thermometer should be placed in it, and care exercised that the temperature does not exceed 200° C. at any point in the pan of coke.

METHOD OF SAMPLING

(*A*) *For Coal Appearing Dry:*

Sampling Coal, Dry.

3. (*a*) If the sample is coarser than will pass completely a 4760-micron (No. 4) sieve and larger in amount than 10 lb., quickly crush it with the jaw crusher to pass a 4760-micron (No. 4) sieve and reduce it on the larger riffle sampler to 10 lb.;[1] then crush it at once to pass an 840-micron (No. 20) sieve by passing through rolls or an enclosed grinder, and take, without sieving, a 50-g. total moisture sample, immediately after the material has passed through the crushing apparatus. This sample should be taken with a spoon from various parts of the product passing an 840-micron (No. 20) sieve, and should be placed directly in a rubber-stoppered bottle.

(*b*) Thoroughly mix the main portion of the sample, reduce on the small riffle sampler to about 200 g., and pulverize to pass a 250-micron (No. 60) sieve by any suitable apparatus without regard to loss of moisture. After all the material has been passed through the 250-micron (No. 60) sieve, mix and divide it on the small riffle sampler to about 50 g. Transfer the final sample to a 4-oz. rubber-stoppered bottle. Determine moisture in both the 250-micron (No. 60) sieve sample and the 840-micron (No. 20) sieve sample in accordance with Sections 6, 7, and 8 under the Determination of Moisture.

(*c*) *Calculation.*—Calculate the analysis of the coal passing the 250-micron (No. 60) sieve which has become partly air-dried during sampling, to the dry-coal basis, by dividing each result by 1 minus its content of moisture. Compute the analysis of the coal "as received" from the dry-coal analysis by multiplying by 1 minus the total moisture found in the sample passing an 840-micron (No. 20) sieve.

(*B*) *For Coal Appearing Wet:*

Sampling Coal, Wet.

4. (*a*) Spread the sample on tared pans, weigh, and air-dry at room temperature, or in the special drying oven, shown in Fig. 1, at 10 to 15° C. above room temperature, and weigh again. The drying should be continued until the loss in weight is not more than 0.1 per cent per hour. Complete the sampling as described in Section 3 for dry coal.

(*b*) *Calculation.*—Correct the moisture found in the air-dried sample passing an 840-micron (No. 20) sieve to total moisture "as received," as follows:

$$\frac{100 - \text{percentage of air-drying loss}}{100} \times (\text{percentage of moisture in air-dried sample passing an 840-micron (No. 20) sieve}) + (\text{percentage of air-drying loss}) = (\text{total moisture "as received"})$$

[1] If the sample is crushed to pass a 3360-micron (No. 6) sieve it may be reduced to 5 lb.

(*c*) Calculate the analysis to "dry-coal" and "as-received" bases as described in Section 3 for dry coal, using for the "as-received" calculation the total moisture as found by the formula in Paragraph (*b*) in place of the moisture found in the coal passing an 840-micron (No. 20) sieve.

NOTES

Freshly mined or wet coal loses moisture rapidly on exposure to the air of the laboratory, hence the sampling operations between opening the container and taking the total-moisture sample passing an 840-micron (No. 20) sieve must be conducted with the utmost dispatch and with minimum exposure to air.

The accuracy of the method of preparing laboratory samples should be checked frequently by resampling the rejected portions and preparing a duplicate sample. The ash in the two samples should not differ more than the following limits:

No carbonates present	0.4 per cent
Considerable carbonate and pyrite present	0.7 "
Coals with more than 12 per cent ash, containing considerable carbonate and pyrite	1.0 "

(*C*) *For Coke:*

Sampling Coke.

5. (*a*) *Total Moisture Determination.*—Dry the entire sample received at the laboratory, without any preliminary crushing, to constant weight at a temperature of not less than 104 nor more than 200° C.[1]

Calculate the loss in weight to percentage of moisture, which shall constitute the total moisture in the coke as received at the laboratory.

The allowable difference in duplicate determinations by the same analyst is 0.5 per cent.

(*b*) *Reduction of Sample.*—Crush the dried sample mechanically with a jaw or roll crusher, or by hand on a chilled iron or hard-steel plate by impact of a hard bar or sledge, avoiding all rubbing action, as otherwise the ash content will be materially increased by the addition of iron from the sampling apparatus, even though hardened iron or steel is used. Continue the crushing until all the sample passes through a 4760-micron (No. 4) sieve, mix and reduce on the large riffle sampler to not less than 5 lb.; again crush the 5-lb. sample sufficiently fine to pass an 840-micron (No. 20) sieve; mix and reduce on the small riffle sampler to 200 g. Transfer this 200-g. portion to the porcelain jar of an Abbé ball mill and pulverize to pass a 250-micron (No. 60) sieve. When pulverization is complete,

[1] Experiments made at the U. S. Bureau of Mines have shown that results checking within 0.5 per cent are obtained between these temperature limits. See A. C. Fieldner and W. A. Selvig, "The Determination of Moisture in Coke," U. S. Bureau of Mines, *Technical Paper No. 148* (1917).

pour the contents of the jar on a $\frac{1}{2}$-in. screen and separate the sample from the pebbles by shaking the screen. Pass all the material through a 250-micron (No. 60) sieve, pulverizing any coarse particles by impact in a hard-steel diamond mortar, and mix with the remainder of the sample. Reduce the sample through the small riffle sampler to about 50 g. and transfer to a rubber-stoppered glass bottle.

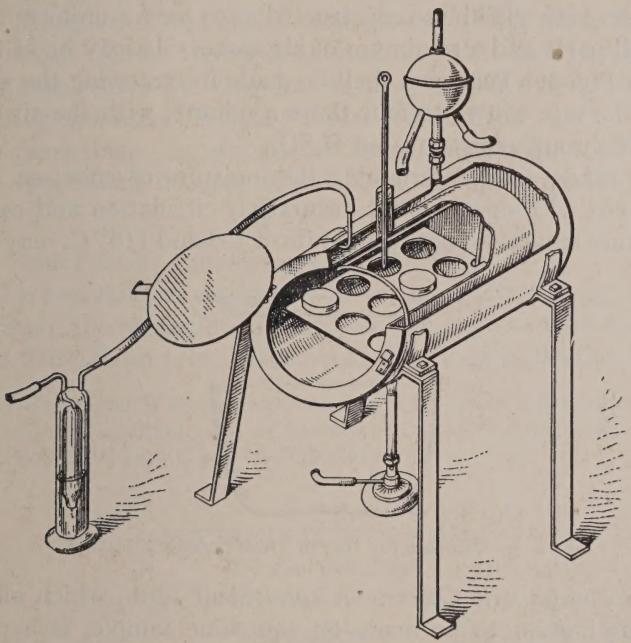


FIG. 3.—Toluene or Glycerin and Water Oven for Determining Moisture (*Technical Paper No. 8*, Bureau of Mines, p. 8 (1926)).

(*c*) In case a ball mill is not available for fine grinding, quarter the 5-lb. sample passing an 840-micron (No. 20) sieve to 200 g. and pulverize to pass a 250-micron (No. 60) sieve by means of a hard-steel roll crusher in which the rolls revolve at the same speed, or by impact in a hard-steel diamond mortar. The use of rubbing surfaces such as a disk pulverizer or a bucking board is never permissible for grinding coke.

NOTES

The accuracy of the method of preparing laboratory samples should be checked frequently by resampling the rejected portions and preparing a duplicate sample. The ash in the two samples should not differ more than 0.4 per cent.

METHODS OF ANALYSIS

DETERMINATION OF MOISTURE

(For Coal and Coke)

APPARATUS

Apparatus for Moisture Determination.

6. (*a*) *Moisture Oven* (*for coal*).—For determining the moisture of coal, the oven shall be so constructed as to have a uniform temperature in all parts and a minimum of air space. It may be of the form shown in Fig. 3. Provision shall be made for renewing the air in the oven at the rate of two to four times a minute, with the air dried by passing it through concentrated H_2SO_4.

(*For coke*).—For determining the moisture of coke, an ordinary drying oven with openings for natural air circulation and capable of temperature regulation between limits of 104 and 110° C. may be used.

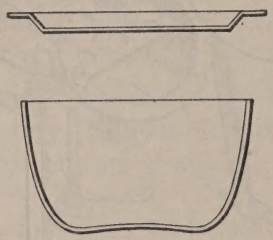


FIG. 4.—Capsule for Use in Determining Moisture.

(*b*) *Capsules with Covers.*—A convenient form, which allows the ash determination to be made on the same sample, is a porcelain capsule, $\frac{7}{8}$ in. deep and $1\frac{3}{4}$ in. in diameter; or a fused silica capsule of similar shape. This shall be used with a well-fitting flat aluminum cover, illustrated in Fig. 4.

Platinum crucibles or glass capsules with ground-glass caps may also be used. They should be as shallow as possible, consistent with convenient handling.

METHOD

(*A*) *For Coal or Coke Passing 250-micron* (*No. 60*) *Sieve:*

Determination of Moisture.

7. (*a*) Heat the empty capsules under the conditions at which the sample is to be dried, place stopper or cover on capsule, cool over concentrated H_2SO_4 (sp. gr. 1.84) for 30 minutes, and weigh. Dip out with a spoon or spatula from the sample bottle approximately 1 g. of the sample; put this quickly into the capsule, close, and weigh at once.

(*b*) An alternate procedure (more open to error), after transferring an amount slightly in excess of 1 g., is to bring to exactly 1 g. in weight (±0.5 mg.) by quickly removing the excess weight of the sample with a spatula. The utmost dispatch must be used in order to minimize the exposure of the sample until the weight is found.

After removing the covers, quickly place the capsules in a preheated oven (at 104 to 110° C.) through which passes a current of air dried by concentrated H_2SO_4 (the current of dry air is not necessary for coke). Close the oven at once and heat for 1 hour. Then open the oven, cover the capsules quickly and place them in a desiccator over concentrated H_2SO_4. When cool, weigh.

The percentage of moisture in the sample passing a 250-micron (No. 60) sieve shall be used to calculate the other results to a dry basis.

(*B*) *For Coal Passing 840-micron (No. 20) Sieve:*

8. Use 5-g. samples, weighed with an accuracy of 2 mg., and heat for 1½ hours; the procedure is otherwise the same as with the sample passing a 250-micron (No. 60) sieve as described in Section 7.

NOTE.—For methods of greater accuracy see Report of Sub-Committee II on Moisture, of the Joint Committee on Analysis of Coal, *Proceedings*, Am. Soc. Testing Mats., Vol. XIV, Part I, pp. 419–423 (1914).

DETERMINATION OF ASH
(For Coal and Coke)

APPARATUS

Apparatus for Ash Determination.

9. (*a*) *Gas or Electric Muffle Furnace (for Coal).*—For determination of ash of coal, the muffle shall have good air circulation and be capable of having its temperature regulated between 700 and 750° C.

(*b*) *Gas or Electric Muffle Furnace or Meker Burner (for Coke).*—For determination of ash of coke, the muffle shall have good air circulation and be capable of having its temperature regulated to not exceed 950° C.

(*c*) *Porcelain Capsules.*—Porcelain capsules, ⅞ in. deep and 1¾ in. in diameter, or similar shallow dishes or platinum crucibles.

METHOD

(*A*) *For Coal:*

Determination of Ash.

10. Place the porcelain capsules containing the dried coal from the moisture determination in a cold muffle furnace, or on the hearth

at a low temperature, and gradually heat to redness at such a rate as to avoid mechanical loss from too rapid expulsion of volatile matter. Finish the ignition to constant weight (±0.001 g.) at a temperature between 700 and 750° C. Cool in a desiccator, and weigh as soon as cold.

NOTES

Before replacing the capsules in the muffle for ignition to constant weight, the ash should be stirred with a platinum or nichrome wire. Stirring once or twice before the first weighing hastens complete ignition.

The result obtained by this method is "uncorrected" ash. For "corrected" ash see the preliminary report.[1] The actual mineral matters in the original coal are usually very different in weight and composition from the weight of the "uncorrected" ash.

(*B*) *For Coke:*

11. Place the capsules containing the dried coke from the moisture determination in a muffle furnace or over a burner, and heat to redness at such a rate as to avoid mechanical loss. Finish the ignition to constant weight (±0.001 g.) at a temperature not exceeding 950° C. Cool in a desiccator and weigh.

NOTES

Before replacing the capsules in the muffle for ignition to constant weight the ash should be stirred with a platinum or nichrome wire. Stirring once or twice before the first weighing hastens complete ignition.

Test the ash for unburned carbon, by moistening it with alcohol; any carbon remaining will show as black particles.

DETERMINATION OF VOLATILE MATTER

(*For Coal and Coke*)

APPARATUS

Apparatus for Volatile Matter Determination.

12. (*a*) *Platinum Crucible with Closely Fitting Cover* (*for Coal*).—The crucible shall be of not less than 10 nor more than 20-cc. capacity; of not less than 25 nor more than 35 mm. in diameter; of not less than 30 nor more than 35 mm. in height.

(*b*) *Platinum Crucible with Closely Fitting Cover* (*for Coke*).—The crucible shall be of 10-cc. capacity, with capsule cover having thin flexible sides fitting down into crucible. Or the double-crucible method may be used, in which the sample is placed in a 10 or 20-cc. platinum crucible, which is then covered with another crucible of such a size that it will fit closely to the sides of the outer crucible, and its bottom will rest $\frac{1}{3}$ to $\frac{1}{2}$ in. above the bottom of the outer crucible.

[1] Report on Fixed Carbon and Ash, *Proceedings*, Am. Soc. Testing Mats., Vol. XIV, Part I, p. 426 (1914).

(c) *Vertical Electric Tube Furnace; or a Gas or Electrically Heated Muffle Furnace (for Coal or Coke).*—The furnace may be of the form as shown in Fig. 5. It shall be regulated to maintain a temperature

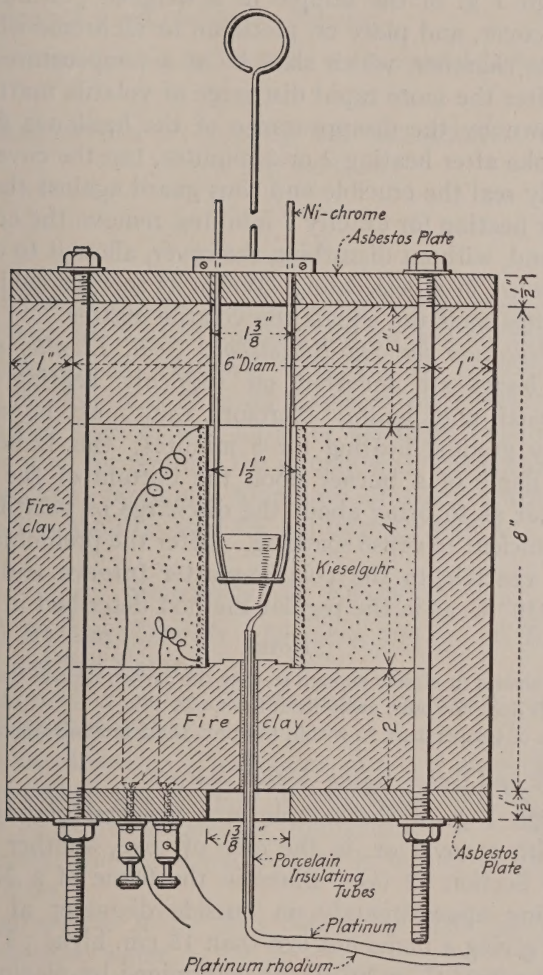


FIG. 5.—Electric Tube Furnace for Determining Volatile Matter. For 110-volt alternating current, 60 ft. of Nichrome Wire, No. 17 B. & S. gage will give the required temperature. The temperature must be controlled by an external resistance. (*Technical Paper No. 8*, Bureau of Mines, p. 11 (1926)).

of 950° C. ±20° C. in the crucible, as shown by a thermocouple kept in the furnace. If the determination of volatile matter is not an essential feature of the specifications under which the coal or coke is bought, a Meker burner may be used.

METHOD

(A) For Coal and Coke, Usual Method:

Determination of Volatile Matter.

13. Weigh 1 g. of the sample in a weighed platinum crucible, close with a cover, and place on platinum or nichrome-wire supports in the furnace chamber, which shall be at a temperature of 950° C. ±20° C. After the more rapid discharge of volatile matter has subsided, as shown by the disappearance of the luminous flame, or in the case of coke after heating 2 or 3 minutes, tap the cover lightly to more perfectly seal the crucible and thus guard against the admission of air. After heating for exactly 7 minutes, remove the crucible from the furnace and, without disturbing the cover, allow it to cool. Coke should be cooled in a desiccator. Weigh as soon as cold. The loss of weight minus moisture equals the volatile matter.

14. *Modification for Sub-Bituminous Coal, Lignite, and Peat.*— Mechanical losses are incurred on suddenly heating peat, subbituminous coal, and lignite; therefore, they must be subjected to a preliminary gradual heating for 5 minutes; this is best done by playing the flame of a burner upon the bottom of the crucible in such a manner as to bring about the discharge of volatile matter at a rate not sufficient to cause sparking. After the preliminary heating, transfer the crucible to the volatile-matter furnace and heat for 6 minutes at 950° C. as in the regular method described in Section 13.

NOTES

The cover should fit closely enough so that the carbon deposit from bituminous and lignite coals does not burn away from the under side.

Regulation of temperature to within the prescribed limits is important.

(B) For Coal and Coke, Using Meker Burner:

15. Weigh 1 g. of the sample in a weighed platinum crucible and close with a cover or, in the case of coke, another crucible as described in Section 12 (*b*). Place in the flame of a No. 4 Meker burner, having approximately an outside diameter at the top of 25 mm. and giving a flame not less than 15 cm. high. The temperature should be 950° C. ±20° C., as determined by placing a thermocouple through the perforated cover, which for this purpose may be of nickel or asbestos. The junction of the couple should be placed in contact with the center of the bottom of the crucible; or the temperature may be indicated by the fusion of pure K_2CrO_4 in the covered crucible (fusion of K_2CrO_4, 940° C.). The crucible shall be placed in the flame about 1 cm. above the top of the burner and the heating continued 7 minutes. Where the gas pressure is variable it is well to use a U-tube attachment to the burner.

DETERMINATION OF FIXED CARBON

(For Coal and Coke)

16. Calculate fixed carbon as follows: **Fixed Carbon Determination.**

100 – (moisture + ash + volatile matter) = percentage of fixed carbon

DETERMINATION OF SULFUR BY THE ESCHKA METHOD

(For Coal and Coke)

APPARATUS

Apparatus for Sulfur Determination.

17. (*a*) *Gas or Electric Muffle Furnace, or Burners.*—For igniting the sample with the Eschka mixture and for igniting the $BaSO_4$.

(*b*) *Porcelain or Platinum Crucibles or Capsules.*—Porcelain capsules, $\frac{7}{8}$ in. deep and $1\frac{3}{4}$ in. in diameter, or porcelain crucibles of 30-cc. capacity, high or low form, or platinum crucibles of similar size shall be used for igniting the sample with the Eschka mixture.

(*c*) Porcelain, platinum, alundum, or silica crucibles of 10 to 15 cc. capacity, shall be used for igniting the $BaSO_4$.

SOLUTIONS AND REAGENTS

Solutions and Reagents.

18. (*a*) *Barium Chloride.*—Dissolve 100 g. of $BaCl_2 \cdot 2H_2O$ in 1000 cc. of distilled water.

(*b*) *Saturated Bromine Water.*—Add an excess of bromine to 1000 cc. of distilled water.

(*c*) *Eschka Mixture.*—Thoroughly mix two parts (by weight) of light calcined MgO and one part of anhydrous Na_2CO_3. Both materials should be as free as possible from sulfur.

(*d*) *Methyl Orange.*—Dissolve 0.02 g. in 100 cc. of hot distilled water and filter.

(*e*) *Hydrochloric Acid.*—Mix 500 cc. of HCl (sp. gr. 1.19), and 500 cc. of distilled water.

(*f*) *Normal Hydrochloric Acid.*—Dilute 80 cc. of HCl (sp. gr. 1.19), to 1 liter with distilled water.

(*g*) *Sodium Carbonate.*—A saturated solution, approximately 60 g. of crystallized or 22 g. of anhydrous Na_2CO_3 in 100 cc. of distilled water.

(*h*) *Sodium-Hydroxide Solution.*—Dissolve 100 g. in 1 liter of distilled water. This solution may be used in place of the Na_2CO_3 solution.

METHOD

Determination of Sulfur.

19. *Preparation of Sample and Mixture.*—Thoroughly mix on glazed paper 1 g. of the sample and 3 g. of Eschka mixture. Transfer to a porcelain capsule, $\frac{7}{8}$ in. deep and $1\frac{3}{4}$ in. in diameter, or a porcelain crucible of 30-cc. capacity, high or low form, or platinum crucible of similar size, and cover with about 1 g. of Eschka mixture.

20. (*a*) *Ignition.*—On account of the amount of sulfur contained in artificial gas, the crucible shall be heated over an alcohol, gasoline or natural gas flame as described in Paragraph (*b*), or in a gas or electrically heated muffle, as described in Paragraph (*c*) for coal and in Paragraph (*d*) for coke. The use of artificial gas for heating the sample and Eschka mixture is permissible only when the crucibles are heated in a muffle.

(*b*) Heat the crucible, placed in a slanting position on a triangle, over a very low flame to avoid rapid expulsion of the volatile matter, which tends to prevent complete absorption of the products of combustion of the sulfur. Heat the crucible slowly for 30 minutes, gradually increasing the temperature and stirring after all black particles have disappeared, which is an indication of the completeness of the procedure.

(*c*) (*For coal*).—Place the crucible in a cold muffle and gradually raise the temperature to 870–925° C. (cherry-red heat) in about 1 hour. Maintain the maximum temperature for about $1\frac{1}{2}$ hours and then allow the crucible to cool in the muffle.

(*d*) (*For coke*).—Place the crucible in a warm muffle and gradually raise the temperature to 870–925° C. (cherry-red heat) in about 30 minutes. Maintain this maximum temperature until on stirring all black particles have disappeared.

(*e*) *Subsequent Treatment.*—Remove and empty the contents into a 200-cc. beaker and digest with 100 cc. of hot water for $\frac{1}{2}$ to $\frac{3}{4}$ hour, with occasional stirring. Filter and wash the insoluble matter by decantation. After several washings in this manner, transfer the insoluble matter to the filter and wash five times, keeping the mixture well agitated. Treat the filtrate, amounting to about 250 cc., with 10 to 20 cc. of saturated bromine water, make slightly acid with HCl and boil to expel the liberated bromine. Make just neutral to methyl orange with NaOH or Na_2CO_3 solution, then add 1 cc. of *N* HCl. Boil again and add slowly from a pipette, with constant stirring, 10 cc. of a 10-per-cent solution of $BaCl_2 \cdot 2H_2O$. Continue boiling for 15 minutes and allow to stand for at least 2 hours, or preferably over night, at a temperature just below boiling. Filter through an ashless filter paper and wash with hot distilled water until a $AgNO_3$

solution shows no precipitate with a drop of the filtrate. Place the wet filter containing the precipitate of $BaSO_4$ in a weighed platinum, porcelain, silica or alundum crucible, allowing a free access of air by folding the paper over the precipitate loosely to prevent spattering. Smoke the paper off gradually and at no time allow it to burn with flame. After the paper is practically consumed, raise the temperature to approximately 925° C. and heat to constant weight.

The residue of MgO, etc., after leaching, should be dissolved in HCl and tested with great care for sulfur. When an appreciable amount is found this should be determined quantitatively. The amount of sulfur retained is by no means a negligible quantity.[1]

(*f*) *Blanks and Corrections.*—In all cases a correction must be applied either (1) by running a blank exactly as described above, using the same amount of all reagents that were employed in the regular determination, or more surely (2) by determining a known amount of sulfate added to a solution of the reagents after these have been put through the prescribed series of operations. If this latter procedure is adopted and carried out, say, once a week or whenever a new supply of a reagent must be used, and for a series of solutions covering the range of sulfur content likely to be met with in the samples, it is only necessary to add to or subtract from the weight of $BaSO_4$ obtained from a sample, whatever deficiency or excess may have been found in the appropriate "check" in order to obtain a result that is more certain to be correct than if a "blank" correction as determined by the former procedure is applied. This is due to the fact that the solubility error for $BaSO_4$, for the amounts of sulfur in question and the conditions of precipitation prescribed, is probably the largest one to be considered. $BaSO_4$ is soluble[2] in acids and even in pure water, and the solubility limit is reached almost immediately on contact with the solvent. Hence, in the event of using reagents of very superior quality or of exercising more than ordinary precautions, there may be no apparent "blank," because the solubility limit of the solution for $BaSO_4$ has not been reached or at any rate not exceeded.

(*g*) Calculate sulfur content as follows:

$$\text{Percentage of Sulfur} = \frac{(\text{Weight of } BaSO_4 - \text{blank}) \times 13.74}{\text{Weight of sample}}$$

NOTE.—As shown in the preliminary report,[3] the Atkinson and sodium-peroxide methods give results in close agreement with the Eschka method. Register[4] has

[1] *Journal*, Am. Chemical Soc., Vol. 21, p. 1125 (1899).
[2] *Journal*, Am. Chemical Soc., Vol. 32, p. 588 (1910); Vol. 33, p. 829 (1911).
[3] *Journal of Industrial and Engineering Chemistry*, Vol. 5, p. 5 (1913).
[4] *Ibid* Vol. 6, p. 812 (1914).

shown that if 5 per cent of nitrogen is present in the gases contained in the bomb calorimeter the sulfur of a sample is almost completely oxidized to H_2SO_4 and the washings of the calorimeter may be used for the determination of sulfur.

DETERMINATION OF PHOSPHORUS IN ASH

(For Coal and Coke)

Method No. 1, for All Cases:

Determination of Phosphorus in Ash.

21. Add to the ash from a 5-g. sample, in a platinum capsule, 10 cc. of HNO_3 and 3 to 5 cc. of HF. Evaporate the liquid and fuse the residue with 3 g. of Na_2CO_3. If unburned carbon is present mix 0.2 g. of $NaNO_3$ with the carbonate. Leach the melt with water and filter the solution. Ignite the residue, fuse with Na_2CO_3 alone, leach the melt and filter the solution. Just acidify with HNO_3 the combined filtrates held in a flask, and concentrate to a volume of 100 cc. Bring the temperature of the solution to 85° C., add 50 cc. of molybdate solution and shake the flask for 10 minutes. If the precipitate does not form promptly and subside rapidly, add enough NH_4NO_3 to cause it to do so. Wash the precipitate six times, or until free from acid, with a 2-per-cent solution of KNO_3, then return it to the flask and titrate with standard NaOH solution. The alkali solution may well be made equal to 0.00025 g. of phosphorus per cubic centimeter, or 0.005 per cent for a 5-g. sample, and is 0.995 of 0.2 *N*.[1] Or the phosphorus in the precipitate may be determined by reduction and titration of the molybdenum with permanganate.

NOTE.—The advantage of the use of HF in the initial attack of the ash lies in the resulting removal of silica. Fusion with alkali carbonate is necessary for the elimination of titanium, which if present, and not removed, will contaminate the phosphomolybdate and is said to sometimes retard its precipitation.

Method No. 2:

22. When titanium is so low as to offer no objection, decompose the ash as described in Section 21, *Method No. 1*, but carry the evaporation only to a volume of about 5 cc. Dilute the solution with water to 30 cc., boil and filter. If the washings are turbid pass them again through the filter. Ignite the residue in a platinum crucible, fuse with a little Na_2CO_3, dissolve the melt in HNO_3 and the solution, if clear, added to the main one. If not clear, filter. From this point proceed as described in Section 21, *Method No. 1*. The fusion of the residue may be dispensed with in routine work on a given sample if it is certain that it is free from phosphorus.

[1] Ulmann and Buch, *Chemical Engineer*, Vol. 10, p. 130 (1900).

DETERMINATION OF FUSIBILITY OF ASH
(For Coal and Coke)

APPARATUS

Apparatus, Fusibility of Ash.

23. (*a*) *Standard Furnace.*—Gas-fired furnaces such as the coal-ash fusion furnace of the Denver Fire Clay Co., the No. 3 Melter's Furnace of the American Gas Furnace Co., or their equivalent shall be used.

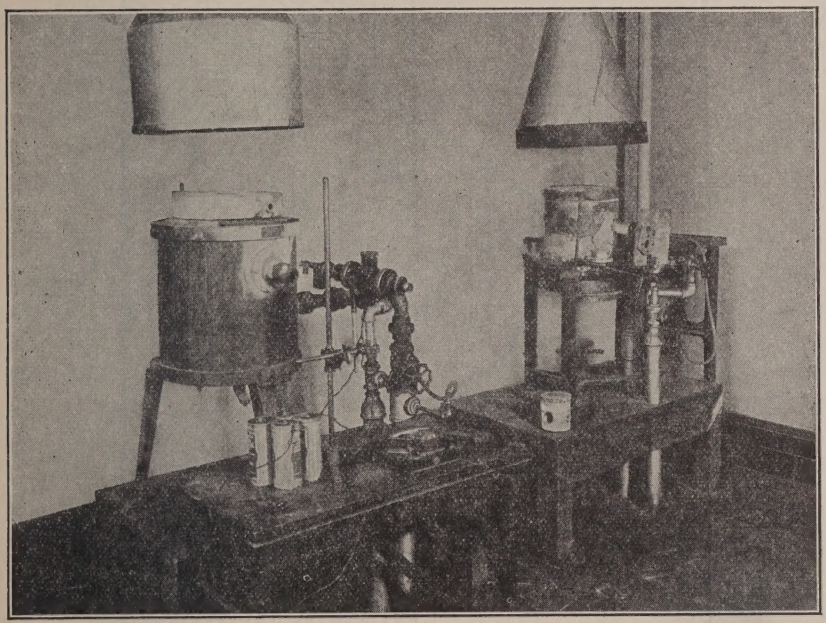


FIG. 6.—Denver Fire-Clay Furnace and No. 3 Melter's Furnace, with Accessories.

These gas-fired furnaces are especially suitable for fusion determination in that the burners are arranged on a tangent near the base of the furnace, thus producing a rotary flame which completely surrounds the crucible in which the ash cones are placed. The whirling flame heats the crucible uniformly and when the furnaces are operated with an excess of gas over air, a reducing atmosphere is maintained in the crucible, which condition gives the lowest point at which the ash fuses. Either natural or artificial gas may be used. Air should be supplied at uniform pressure.

The stock design of the No. 3 Melter's Furnace should be modified by providing the upper cylinder with two holes in the side; a 2-in.

observation hole with its center 4 in. from the top of the cylinder (excluding cover plate), and a 1-in. thermocouple hole 90 deg. to the right of the observation hole; the bottoms of these holes being in the same horizontal plane. The coal-ash fusion furnace as furnished by the Denver Fire Clay Co. is provided with suitable observation and thermocouple holes. The interior of the furnaces are cylindrical and approximately 7 in. in diameter. Counter-balanced sheet-iron cano-

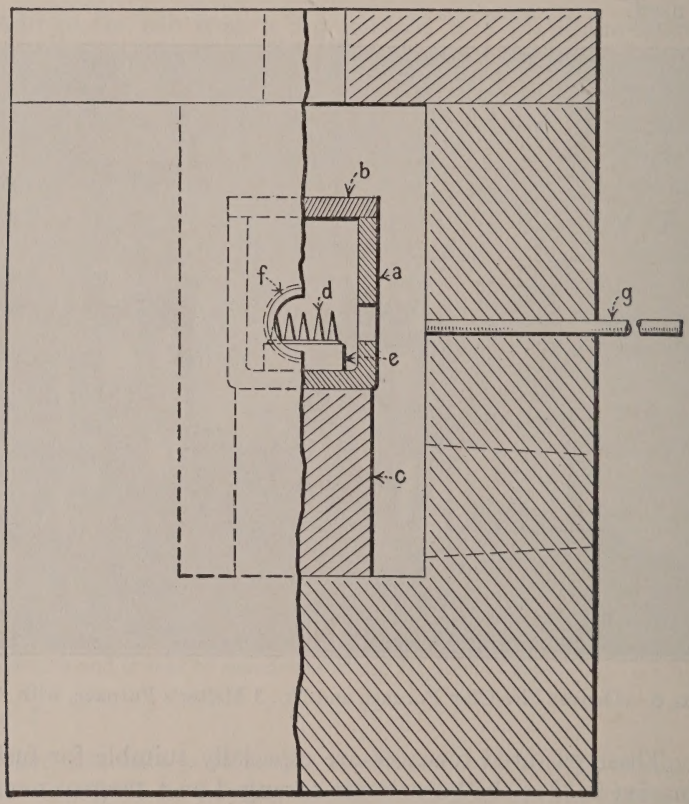


FIG. 7.—Section of Furnace Arranged for Fusion Tests.

pies connected with telescopic flues to an exhaust system are placed over the furnaces for conducting the hot gases out of the room. Two furnaces and accessories are shown in Fig. 6.

(*b*) *Refractory Crucibles.*—The interior of the fusion furnace as arranged for making a test is shown in Fig. 7. Crucible *a* is a Corundite crucible about 3 in. in inside diameter and about 4½-in. in height outside, with a wall thickness of about ½ in. The crucible

is provided with a cover, *b*, and shall have two holes in the side, an observation hole 2 in. in diameter and a thermocouple hole 1 in. in diameter, 90 deg. to the right of the observation hole. The bottoms of the two holes shall be in the same horizontal plane. The crucible is supported by a Corundite support, *c*, so that the two holes in the crucible are in line with the corresponding holes in the furnace wall. The ash cones, *d*, are supported on Corundite disk *e* so that they are visible through the observation hole of the crucible and furnace wall.

(*c*) *Observation Hole.*—A fused silica, alundum, or refractory porcelain tube, *f*, $1\frac{7}{8}$ in. in external diameter and 6 to 7 in. long, is

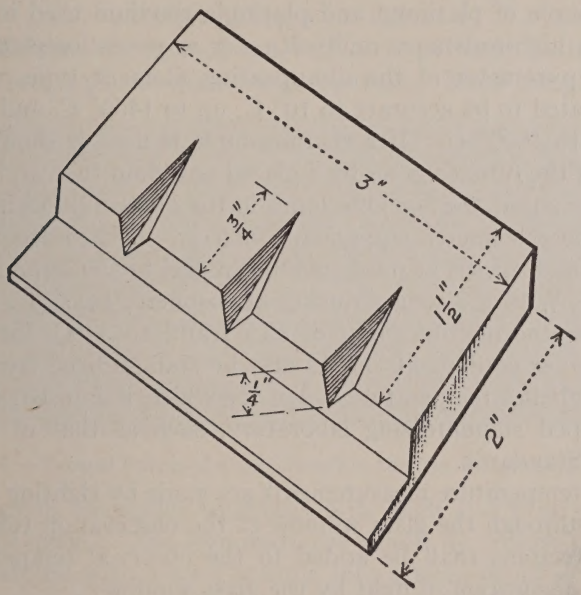


FIG. 8.—Brass Cone Mold.

cemented in the 2-in. observation hole of the furnace, the inner end being flush with the inside furnace wall and the other end projecting out of the furnace. A brass sleeve carrying a single thin glass window, made from ordinary soda glass, shall be slipped on the outer end of the observation tube to prevent the escape of burning gas, which would interfere with a convenient observation of the cones. When observations at high temperatures are made, the cones should be viewed through a piece of colored glass.

(*d*) *Blowing Tube.*—At furnace temperatures above 1000° C. it is very difficult to observe the ash cones. An open refractory porcelain blowing tube, *g*, Fig. 7, about $\frac{1}{4}$ in. in inside diameter shall be

inserted in the thermocouple hole, the inner end being flush with the inside of the furnace wall. The blowing tube shall be connected to the compressed air line by means of rubber tubing and the air shall be let into the tube at the time of observation by means of a pinch-cock, thus momentarily cooling the ash cones and rendering them visible.

(*e*) *Brass Cone Mold.*—A brass cone mold for making ash cones, $\frac{3}{4}$ in. high and $\frac{1}{4}$ in. at each side of the base, which is an equilateral triangle, is illustrated in Fig. 8.

(*f*) *Pyrometer.*[1]—Temperature measurements shall be made with a thermocouple of platinum and platinum-rhodium used in conjunction with a high-resistance millivoltmeter or potentiometer; or with an optical pyrometer of the disappearing filament type, which has been calibrated to be accurate to 10° C. up to 1400° C. and to 15° C. from 1400 to 1600° C. If a thermocouple is used it should be protected from the furnace gases by a glazed porcelain tube and so placed in the furnace that the hot junction is in the immediate vicinity of the cones. The pyrometer equipment shall be checked frequently by mounting small pieces of pure gold and nickel in the same manner as the cones. With a strong reducing atmosphere, 1452° C. should be obtained for the melting point of nickel and 1063° C. for the gold. The pyrometer equipment should also be standardized from time to time through the temperature range for which it is used, by a suitably equipped standardizing laboratory, such as that of the U. S. Bureau of Standards.

(*g*) If temperature measurements are made by sighting an optical pyrometer through the glass window of the observation tube the following corrections shall be added to the observed temperatures to correct for absorption of light by the glass window:

CORRECTION TO OBSERVED TEMPERATURE FOR ABSORPTION OF LIGHT BY A SINGLE CLEAN GLASS WINDOW[2]

OBSERVED TEMPERATURE, DEG. CENT.	CORRECTION TO BE ADDED, DEG. CENT.
800	5
1000	8
1200	10
1400	13
1600	16

[1] For detailed information concerning the use of pyrometers see "Pyrometric Practice," U. S. Bureau of Standards, *Technologic Paper No. 170* (1921).

[2] U. S. Bureau of Standards *Technologic Paper No. 170*, p. 117 (1921).

PREPARATION OF ASH

Preparation of Ash.

24. Spread out 50 to 100 g. of the sample passing a 250-micron (No. 60) sieve on a 6-in. fire-clay roasting dish, and completely convert to ash in a muffle furnace at a temperature of 800 to 900° C. Transfer 5 to 10 g. of this ash to an agate mortar[1] and grind to pass a 74-micron (No. 200) sieve. Then place the ash in a silica or porcelain capsule, $\frac{5}{8}$ in. deep and $1\frac{3}{4}$ in. in diameter, and ignite for a period of two hours in a current of oxygen, at a temperature of 800 to 850° C. This ignition is made to insure complete and uniform oxidation of the ash.

PREPARATION OF CONES

Preparation of Cones.

25. Moisten the ignited ash with a 10-per-cent dextrin solution and work into a plastic mass with a spatula. Mold the plastic material into small triangular pyramids $\frac{3}{4}$ in. high and $\frac{1}{4}$ in. wide at the side of the base. The pyramids are made by firmly pressing the plastic material with a steel spatula into a brass mold of the dimensions mentioned, the mold being similar to that shown in Fig. 8. Strike off the surface smooth and remove the cone from the mold by applying a small knife blade at the base. Mount the cones when dry in a refractory base composed of a mixture of equal parts of kaolin and calcined alumina. Moisten the base mixture to make it workable, and spread a part of it out on a sheet-iron plate. Then mount the cone in a vertical position in a small hole made in the base, and put base material into the hole around the bottom of the cone to fill the crevices and make the cone stand firmly. Usually five cones are mounted in one base in the manner shown in Fig. 9. Dry the sheet-iron plate with the test piece on a hot plate. Ignite the cones at a dull red heat for 30 minutes in an open muffle to remove the carbonaceous material.

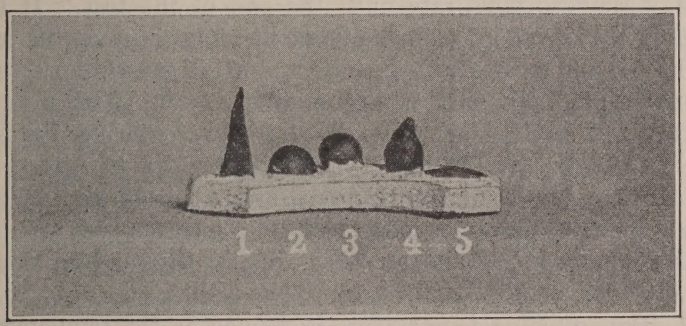


FIG. 9.—Typical Forms of Cones Fused in the No. 3 Melter's Furnace.

[1] A mechanical agate-mortar grinder will save time where many determinations are made.

METHOD OF HEATING

Heating of Cones.

26. Place the test piece in the muffle crucible of the furnace in the position shown in Fig. 7, place the loosely fitting cover *b*, on the crucible, and ignite the gas. It is necessary to let the gas burn about 10 minutes to heat the furnace parts before the large cover plate of the furnace is replaced; otherwise the flame is apt to blow out. During this time increase the flow of gas and air sufficiently to cause the combustion to take place just above the tuyeres and yet maintain a yellowish flame at least 6 in. above the opening in the furnace cover plate. While such a flame is maintained above the furnace, gradually increase the temperature by a suitable adjustment of gas and air to 800° C., then reduce the rate of heat increase to not less than 5° C. and not more than 10° C. per minute. Maintain this rate until the end of the test. It is also important that the 6-in. reducing flame be maintained at the furnace vent throughout the test, if possible, and at all events up to a temperature of 1450° C. Temperatures above 1450° C. require larger proportions of air to gas; however, a strongly reducing atmosphere is not so essential at the higher temperatures, as refractory ashes, owing to their low iron-oxide content, are only slightly affected by oxidizing or reducing atmospheres. After a test has been completed, turn the supply of gas and air off gradually to avoid cracking the muffle crucible.

Softening Temperature.

27. The softening temperature is defined as the temperature at which the cone has fused down to a spherical lump, as shown in cones 2 and 3 of Fig. 9. Cone 4 has almost reached the softening temperature.

NOTES.

Critical points other than the softening temperature, that may be observed during the test, and which may be of value are as follows:

The Initial Deformation Temperature.—The temperature at which the first rounding or bending of the apex of the cone takes place, as shown in cone 1 of Fig. 9. Such bending must not be confused with a shrinking or warping of the cone.

The Fluid Temperature.—The temperature at which the cone has spread out over the base in a flat layer, as represented by cone 5 of Fig. 9.

ULTIMATE ANALYSIS

(For Coal and Coke)

CARBON AND HYDROGEN

Method With Gas-Heated Combustion Furnace:

Carbon and Hydrogen Determination.

28. The determination of carbon and of hydrogen shall be made with a weighed quantity of the sample in a 25-burner combustion

furnace of the Glaser type. The products of combustion shall be thoroughly oxidized by passing them over red-hot CuO and $PbCrO_4$, and shall be fixed by absorbing the water in a weighed Marchand tube filled with granular $CaCl_2$ and by absorbing the CO_2 in a Liebig bulb containing a 30-per-cent solution of KOH.

Apparatus.

29. *Apparatus.*—The apparatus used shall consist of a purifying train in duplicate, a combustion tube in the furnace, and an absorption train.

Purifying Train.

(*a*) The purifying train shall consist of the following purifying reagents arranged in order of passage of air and oxygen through them: H_2SO_4, KOH solution, soda lime, and granular $CaCl_2$. One of the trains is for air and one for oxygen. In the H_2SO_4 and KOH scrubbing bottles the air and the oxygen shall be made to bubble through about 5 mm. of the purifying reagent. Both purifying trains shall be connected to the combustion tube by a Y-tube, the joint being made tight by a rubber stopper.

Combustion Tube.

(*b*) The combustion tube shall be made of hard Jena glass, or its equivalent. Its external diameter shall be about 21 mm., and its total length 1 meter. The first 30 cm. of the tube shall be empty; following this empty space is an asbestos plug (acid-washed and ignited) or in its place a roll of oxidized copper gauze may be used; the next 40 cm. shall be filled with "wire" CuO; a second asbestos plug separates the copper oxide from 10 cm. of fused $PbCrO_4$, which shall be held in place by another asbestos plug 20 cm. from the end of the tube. The end of the tube shall be drawn out for rubber-tubing connection with the absorption train.

Absorption Train.

(*c*) The absorption train shall consist of a Marchand tube filled with granular $CaCl_2$ to absorb moisture. The $CaCl_2$ should be saturated with CO_2 before using. The Marchand tube shall be followed by a Liebig bulb containing a 30-per-cent KOH solution, in which any possible impurities, as ferrous iron or nitrates, have been oxidized by a little $KMnO_4$. A guard tube, containing granular $CaCl_2$ and soda lime, shall be attached to the Liebig bulb to absorb any CO_2 escaping the KOH solution and any water evaporating from that solution.

Procedure.

30. *Procedure.*—Connect the train to an aspirator which draws the products of combustion through the entire train. A guard tube of $CaCl_2$ prevents moisture from running back into the absorption train. Maintain the suction constant by a Mariotte flask. The advantage of aspirating the gases through the train rather than forcing them through by pressure is that the pressure on the rubber connections is from the outside, so that gas-tight connections are

more easily maintained than if the pressure is on the inside of the tube. Make the connections as tight as possible. The usual test for tightness is to start aspiration at the rate of about three bubbles of air per second through the potash bulb, and then to close the inlet for air and oxygen at the opposite end of the train; if there is no more than one bubble per minute in the potash bulb, the apparatus shall be considered tight.

Before starting a determination when the train has been idle some hours, or after any changes in chemicals or connections, run a blank by aspirating about 1 liter of air through the train, which is heated in the same manner as if a determination on the sample were being made. If the Liebig bulb and the tube containing calcium chloride show a change in weight of less than 0.5 mg. each, the apparatus is in proper condition for use.

Use a porcelain or platinum boat provided with a glass weighing tube of suitable size, which is fitted with an accurately ground glass stopper. Weigh the tube and empty boat. Quickly place in the boat approximately 0.2 g. of the air-dry sample (passing a 250-micron (No. 60) sieve and finer, or better, passing a 149-micron (No. 100) sieve if much free impurity is present). Place the boat at once in the weighing tube, which is quickly stoppered to prevent moisture change in the sample while weighing, and transfer to the furnace. Connect the absorption tubes and transfer the boat and sample from the weighing tube to the combustion tube, which should be cool for the first 30 cm. The CuO should be red hot and the $PbCrO_4$ at a dull-red heat. Transfer the boat from the weighing tube to the combustion tube as rapidly as possible.

As soon as the boat is in place near the asbestos plug at the beginning of the copper oxide insert the stopper connecting with the purifying train and start the aspiration with pure oxygen gas at the rate of three bubbles per second. Turn on one burner about 10 cm. back from the boat, and continue the aspiration carefully until practically all the moisture is expelled from the sample. Then increase the heat very gradually until all the volatile matter has been driven off. In driving off the volatile matter the heat must be applied gradually in order to prevent a too rapid evolution of gas and tar, which may either escape complete combustion or may be driven back into the purifying train. Increase the heat slowly by turning on more burners under the open part of the tube until the sample is ignited; then the temperature may be increased rapidly, but care should be taken not to melt the combustion tube.

Any moisture collecting in the end of the combustion tube or

in the rubber connection joining it to the $CaCl_2$ tube may be driven over into the $CaCl_2$ tube by carefully warming with a piece of hot tile. Continue the aspiration with oxygen for 2 minutes after the sample ceases to glow, then turn off the heat and aspirate about 1200 cc. of air. Disconnect the absorption bulbs, wipe with a clean cloth, and allow to cool to the balance-room temperature before weighing.

Calculate the percentages of hydrogen and carbon as follows:

$$\text{Percentage of hydrogen} = \frac{11.19 \times (\text{increase in weight of } CaCl_2 \text{ tube})}{\text{Weight of sample}}$$

$$\text{Percentage of carbon} = \frac{27.27 \times (\text{increase in weight of KOH bulb})}{\text{Weight of sample}}$$

Weigh the ash in the boat and carefully inspect it for any unburned carbon, which would destroy the value of the determination.

Method with Electrically-Heated Combustion Furnace:

31. (*a*) *Apparatus.*—An electrically-heated combustion furnace of the Heraeus type is used by the Bureau of Mines.[1] It consists of three independent heaters, two of which are provided with sheave wheels, and are mounted on a track so that they are movable along the tube; the third heater which surrounds the $PbCrO_4$, is stationary. **Apparatus.**

(*b*) The furnace as provided by the manufacturer does not include the small stationary heater. This may be made in the laboratory by winding an alundum tube 12 cm. in length with No. 20 nichrome II wire and enclosing it in a cylinder packed with magnesia-asbestos. The movable heaters have very thin platinum foil, weighing about 9 g. in all, wound on a porcelain tube of 30 mm. internal diameter. The larger one which heats the CuO, is 350 mm. in length, and the smaller one, which heats the sample in the boat, is 200 mm. in length. **Furnace.**

(*c*) The Jena glass or fused silica combustion tube, of about 21 mm. external diameter and 900 mm. in length, is supported by an asbestos-lined nickel trough. The current through each heater shall be regulated independently by separate rheostats, mounted on the frame of the furnace. The two platinum-wound heaters require an average current of about 4.5 amperes at a pressure of 220 volts, although for heating rapidly a larger amperage is necessary. **Combustion Tube.**

32. *Procedure.*—Purify the oxygen or air entering the combustion tube by passing through a Tauber's drying apparatus, which contains the following reagents arranged in order of the passage of **Procedure.**

[1] U. S. Bureau of Mines, *Technical Paper No. 8*, p. 18 (1926).

air or oxygen through them: H_2SO_4, for removing possible traces of ammonia, 30-per-cent KOH solution, granular soda lime, and granular $CaCl_2$. Connect one side of the train directly to a Linde oxygen tank provided with a reducing valve for regulating the oxygen pressure; use the other side of the train for purifying the air supply.

The absorption train shall consist of a 5-in. U-tube, filled with granular $CaCl_2$ to absorb moisture. Before using, saturate the $CaCl_2$ with CO_2 to avoid possible absorption of CO_2 during a determination by any traces of CaO that may be present. This saturating is done most conveniently by placing a quantity of $CaCl_2$ in a large drying jar, and filling the jar with CO_2. After standing over night, draw dry air through the jar to remove the CO_2. Keep the treated $CaCl_2$ in well-stoppered bottles.

Connect the $CaCl_2$ tube to a Vanier potash bulb containing a 30-per-cent KOH solution and granular $CaCl_2$. Six to eight determinations may be made without recharging this bulb. Connect the potash bulb to an aspirator through a guard tube containing granular $CaCl_2$ and soda lime, and a Mariotte flask. The Mariotte flask keeps the pressure constant.

In general, the method of determination is the same as the one used with the gas furnace as described in Sections 29 and 30. By moving the heaters toward the end of the tube where the gases enter, and cutting in the electric current, the air may be warmed enough to thoroughly dry the tube and its contents. Cut off the current from the small heater, and move the large heater over the CuO; keep exposed about 250 mm. of that part of the combustion tube between the two heaters where the boat containing the sample is to be placed. Turn the full current on the large heater to bring the CuO to a red heat. When this temperature is reached it is necessary to reduce the current with the rheostat to avoid melting the tube. In the meantime, weigh and connect the absorption train and place the boat containing the sample in the exposed and cooler part of the tube between the two heaters.

Pass the current through the shorter heater. By manipulating the rheostat and by gradually pushing this heater toward the boat, the rate of evaporation of moisture and evolution of volatile matter may be readily controlled.

After combustion is complete, turn the electric current off the smaller heater and move this heater back, to allow the tube to cool for the next determination. Conduct the final aspiration of air and the weighing of the absorption train as described in Sections 29 and 30 under the gas-furnace method.

NOTE

In place of granulated $CaCl_2$, concentrated H_2SO_4 may be used for collecting the water formed by combustion. In such cases the air and oxygen entering the combustion tube and the gas leaving the potash bulb must also be dried by H_2SO_4.

Other suitable forms of absorption vessels than those indicated in the above procedure may be used.

NITROGEN

Nitrogen Determination.

33. The Kjeldahl-Gunning method is recommended for the determination of nitrogen. This method has the advantage over either the simple Kjeldahl or the Gunning method, in requiring less time for the complete oxidation of the organic matter, and in giving the most uniform results.

The Kjeldahl-Gunning Method.—Boil 1 g. of the sample with 30 cc. of concentrated H_2SO_4, 7 to 10 g. of K_2SO_4, and 0.6 to 0.8 g. of metallic mercury in a 500-cc. Kjeldahl flask until all particles of the sample are oxidized and the solution nearly colorless. Continue the boiling at least 2 hours after the solution has reached the straw-colored stage. The total time of digestion will be from 3 to 4 hours except in the case of coke and anthracite coal which require much longer digestion. The addition of a few crystals of $KMnO_4$, after the solution has cooled enough to avoid violent reaction, tends to insure complete oxidation. Coke and anthracite coal should be ground to an impalpable powder, as they are very difficult to oxidize. Even if this is done the digestion may require 12 to 16 hours.

After cooling, dilute the solution to about 200 cc. with cold water. If the dilution with water has warmed the solution, cool it again and add the following reagents: 25 cc. K_2S solution (40 g. K_2S per liter) to precipitate the mercury, 1 to 2 g. of granular zinc to prevent bumping, and finally enough strong NaOH solution (usually 80 to 100 cc.) to make the solution distinctly alkaline. The danger of loss of NH_3 may be minimized by holding the flask in an inclined position while the NaOH solution is being added. The alkaline solution runs down the side of the flask and forms a layer below the lighter acid solution. After adding the alkaline solution, connect the flask at once to the condensing apparatus and mix the solution by gently shaking the flask.

Distill the NH_3 over into a measured amount (10 cc.) of standard H_2SO_4 solution, to which has been added sufficient cochineal indicator for titration. Care should be taken that the glass connecting tube on the end of the condenser dips under the surface of the standard acid. Distill the solution slowly until 150 to 200 cc. of distillate has passed over. To avoid mechanically entrained alkali passing over

into the condenser, the rate of distillation should not exceed 100 cc. per hour. Titrate the distillate with standard NH_3 solution (20 cc. NH_4OH solution = 10 cc. H_2SO_4 solution = 0.05 g. nitrogen). Standard NaOH or KOH solution with methyl orange or methyl red as indicator may be used instead of NH_3 and cochineal.

A blank determination shall be made in exactly the same manner as described above, except that 1 g. of pure sucrose (cane sugar) shall be substituted in place of the sample. The nitrogen found in this blank determination shall be deducted from the result obtained with the sample.

The K_2S and NaOH may be dissolved in a single stock solution. Dissolve sufficient K_2S in the water before adding the NaOH, to make a solution in which the quantity necessary for a nitrogen determination (80 to 100 cc.) contains 1 g. of K_2S. Twelve grams of K_2S and 500 g. of NaOH in one liter of water, are required for the above proportions.

OXYGEN

Oxygen Determination.

34. There being no satisfactory direct method of determining oxygen, it shall be computed by subtracting the sum of the percentages of hydrogen, carbon, nitrogen, sulfur, water and ash from 100. The result so obtained is affected by all the errors incurred in the other determinations and especially by the change in weight of the ash-forming constituents on ignition. In the case of coke, iron pyrite changes to ferric oxide, increasing the ash and causing a negative error in the oxygen equivalent to three-eighths of the pyritic sulfur. On the other hand, there is always a loss on ignition, of water of composition from the clayey and shaley constituents, CO_2 from carbonates, etc., which tends to compensate the absorption of oxygen.

Corrected Oxygen Value.

35. *Corrected Oxygen (for Coal).*—When a more correct oxygen value is desired, it may be obtained by making the corrections indicated in the following formula:

$$\text{Corrected oxygen} = 100 - [\,(C - C') + (H - H') + N + H_2O + S' + \text{corrected ash}];$$

where C = total carbon;
C' = carbon of carbonates;
H = total hydrogen less hydrogen of water;
H' = hydrogen from water of composition in clay, shale, etc.,
N = nitrogen;
H_2O = moisture as found at 105° C.;
S' = sulfur not present as pyrite or sulfate. This is usually small. In many types of coal it may be disregarded.

Corrected ash = mineral constituents originally present in the coal. For most purposes this can be determined with sufficient accuracy by adding to the ash, as found, five-eights of the weight of pyritic sulfur, the CO_2 of carbonates and the water of composition of clay, shale, etc. See also Determination of Ash, Sections 9 and 10.

CALORIMETRIC DETERMINATION

(For Coal and Coke)

APPARATUS

Apparatus, Calorimetric Determination.

36. (*a*) *Combustion Bombs.*—The Atwater, Davis, Emerson, Mahler, Parr, Peters, Williams, or similar bombs may be used. The bomb shall have an inner surface of platinum, gold, porcelain enamel, or other material which is not attacked by HNO_3 and H_2SO_4, or other products of combustion.

(*b*) *Calorimeter Jacket.*—The calorimeter shall be provided with a water-jacket having a cover to protect the calorimeter from air currents. The jacket shall be kept filled with water within 2 or 3° C. of the temperature of the room (except in calorimeters which are totally submerged, where the jacket temperature is controlled by a thermostat) and should be stirred continuously by some mechanical stirring device.

(*c*) *Stirring of the Calorimeter Water.*—The water in the calorimeter shall be stirred sufficiently well to give consistent thermometer readings while the temperature is rising rapidly. The speed of stirring should be kept constant. A motor-driven screw or turbine stirrer is recommended and the speed should not be excessive. This may be determined by adjusting the temperature of the calorimeter to equality with that of the jacket and allowing the stirrer to run continuously for 10 minutes. If the temperature of the calorimeter rises more than about 0.01° C. in this length of time, the rate of stirring is excessive. Accurate results cannot be obtained when too much energy is supplied by the stirring device or when the rate of stirring is irregular. The portion of the stirring device immersed in the calorimeter should be separated from that outside by non-conducting material, such as hard rubber, to prevent conduction of heat from the motor or outside air.

(*d*) *Thermometers.*—Thermometers used shall have been certified by a government testing bureau and shall be used with the corrections given on the certificate. This shall also apply to electrical resistance or thermo-electric thermometers. Correction shall also be

made for the temperature of the emergent stem of all mercurial thermometers, and for the "setting" of Beckmann thermometers. For accurate work, either Beckmann or special calorimetric thermometers graduated to 0.01 or 0.02° C. are required. Such thermometers should be tapped lightly just before each reading to avoid errors caused by the sticking of the mercury meniscus, particularly when the temperature is falling. A convenient method is to mount a small electric buzzer directly on the top of the thermometer and connect it with a dry cell and a push button. The button should be pressed for a few seconds immediately before each reading.

(*e*) *Oxygen.*—The oxygen used for combustions shall be free from combustible material. The bomb when filled should contain at least 5 per cent of nitrogen to insure complete oxidation of the sulfur.[1] The total amount of oxygen contained in the bomb for combustion shall not be less than 5 g. per gram of the sample. But the combustion must be complete, as shown by the absence of any sooty deposit on opening the bomb after firing.

(*f*) *Firing Wire.*—The sample in the bomb may be ignited by means of either iron or platinum wire. If iron wire is used, it should be of about No. 34 B. & S. gage and not more than 10 cm. (preferably 5 cm.) should be used at a time. A correction of 1600 calories per gram weight of iron wire burned shall be subtracted from the observed number of calories.

(*g*) *Standardization.*—The water equivalent of a calorimeter can best be determined by the use of the standard combustion samples supplied by the U. S. Bureau of Standards. The required water equivalent is equal to the weight of the sample multiplied by its heat of combustion per gram and divided by the corrected rise in temperature.

The calorimeter shall be standardized by the combustion of standard samples supplied by the Bureau of Standards, and used according to the directions given in the certificates which accompany them. A standardization shall consist of a series of not less than five combustions of either the same or different standard materials. The conditions as to the amount of water, oxygen, firing wire, method of correcting for radiation, etc., under which these combustions are made shall be the same as for combustions of the sample. In the case of any disagreement between contracting parties a check standardization may consist of two or more combustions of standardizing samples.

[1] Register, *Journal of Industrial and Engineering Chemistry*, Vol. 16, p. 812 (1914).

PROCEDURE

37. (*a*) *Preparation of Sample.*—The ground sample shall be thoroughly mixed in the bottle and an amount, approximately 1 g., shall be taken out and weighed in the crucible in which it is to be burned. Samples which are likely to be blown out of the crucible should be briqueted. After weighing, the sample should preferably be immediately placed in the bomb and this closed. This procedure is necessary to avoid sublimation in the use of naphthalene for standardization. Procedure.

(*b*) *Preparation of the Bomb.*—The firing wire, if iron, shall be measured and coiled in a small spiral and connected between the platinum terminals, using, if necessary, a piece of platinum wire somewhat heavier than the iron wire, to make the connection. The platinum and the iron shall both be clean. About 0.5 cc. of water should be placed in the bottom of the bomb to saturate with moisture the oxygen used for combustion. When the crucible is put in place in the bomb, the firing wire should touch the sample or briquet of standard material. For the combustion of standardizing samples iron wire is preferable to platinum.

(*c*) *Filling the Bomb with Oxygen.*—Oxygen from the supply tank shall be admitted slowly to avoid blowing the sample from the crucible, and the pressure allowed to reach 20 atmospheres for the larger bombs or about 30 atmospheres for the smaller bombs, so that the bomb shall contain an amount of oxygen sufficient for complete combustion, namely, at least 5 g. per gram of the sample. This method of filling should insure 4 per cent of nitrogen in the larger bombs, irrespective of the nitrogen contained in the oxygen.

(*d*) *Calorimeter Water.*—The calorimeter shall be filled with the required amount of distilled water, depending upon the type of calorimeter. The amount may be determined either by measurement in a standardized flask or by weighing. The amount shall be kept the same as that used in the standardization of the apparatus.

(*e*) *Temperature Adjustments.*—The initial temperature in the calorimeter shall be so adjusted that the final temperature, after the combustion, will not be more than 1° C., preferably about 0.5° C., above that of the jacket, under which conditions the total correction for heat gained from or lost to the surroundings will be small when the rise of temperature is 2 or 3° C., and the effect of evaporation will also be small.

(*f*) *Firing Current.*—The electric current used for firing the charge shall be obtained from storage or dry cells having an electromotive force of not more than 12 volts. A higher voltage is liable

to cause an arc between the firing terminals, introducing additional heat, which cannot be measured with certainty. The circuit should be closed by means of a switch which should remain closed for not more than 2 seconds. When possible, it is recommended that an ammeter be used in the firing circuit to indicate when the firing wire has burned out.

(*g*) *Method of Making an Observation.*—The bomb when ready for firing, shall be placed in the calorimeter, the firing wires connected, the cover put in place and the stirrer and thermometer so placed as not to be in contact with the bomb or container. The stirrer shall then be started and after the thermometer reading has become steady, not less than 2 minutes after the stirrer is started, temperatures shall be read at 1-minute intervals for 5 minutes and the charge then fired, the exact time of firing being noted. Observations of temperature shall then be made at intervals depending upon the method to be used for computing the cooling correction. When the temperature has reached its maximum and is falling uniformly, a series of thermometer readings shall be taken at 1-minute intervals for 5 minutes to determine the final cooling rate.

(*h*) *Titration.*—After a combustion and after allowing the gas to escape the bomb shall be opened and the inside examined for traces of unburned material or sooty deposit. If these are found, the observations shall be discarded. If the combustion appears complete, the bomb shall be rinsed out thoroughly and the washings titrated with a standard alkali solution (1 cc. = 0.02173 g. HNO_3 = 5 calories) using methyl-orange or methyl-red indicator, to determine the amount of acid formed. A correction of 230 calories per gram of HNO_3 should be subtracted from the total heat observed. An additional correction of 1300 calories per gram of sulfur in the sample should be made for the excess of difference in heats of formation of SO_2 and aqueous H_2SO_4 over the heat of formation of aqueous HNO_3.

CALCULATION OF RESULTS

Calculation of Results.

38. The following method of calculation is recommended to take the place of the Pfaundler or other similar formulas for calculating the cooling correction (radiation correction):

Observe (1) the rate of rise, r_1, of the calorimeter temperature in degrees per minute for 5 minutes before firing; (2) the time, a, at

which the last temperature reading is made immediately before firing; (3) the time, b, when the rise of temperature has reached six-tenths of its total amount (this point can generally be determined by adding to the temperature observed before firing, 60 per cent of the expected[1] temperature rise, and noting the time at which this point is reached); (4) the time, c, of a thermometer reading taken when the temperature change has become uniform some 5 minutes after firing; (5) the final rate of cooling, r_2, in degrees per minute for 5 minutes.

Multiply the rate r_1 by the time $b-a$ in minutes and tenths of a minute, and add to this product (subtracted if the temperature was falling at the time a) to the thermometer reading taken at the time a. Multiply the rate r_2 by the time $c-b$ and add this product (subtracted if the temperature was rising at the time c and later) to the thermometer reading taken at the time c. The difference of the two thermometer readings thus corrected, provided the corrections from the certificate have already been applied, gives the total rise of temperature due to the combustion. This multiplied by the water equivalent of the calorimeter gives the total amount of heat liberated. Divide this result, corrected for the heats of formation of HNO_3 and H_2SO_4 observed and for the heat of combustion of the firing wire, when that is included, by the weight of the charge to find the heat of combustion in calories per gram. Calories per gram multiplied by 1.8 give the British thermal units per pound. (See example.)

NOTE.—In practice, the time $b-a$ will be found so nearly constant for a given calorimeter with the usual amounts of fuel that b need be determined only occasionally.

The results shall be reduced to calories per gram or British thermal units per pound of *dry sample*, the moisture being determined upon a sample taken from the bottle at about the same time as the combustion sample is taken.

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[1] When the temperature rise is not approximately known beforehand, it is only necessary to take thermometer readings at 40, 50, 60 seconds (and possibly 70 seconds with some calorimeters) after firing, and from these observations to find when the temperature rise had reached 60 per cent of the total. Thus, if the temperature at firing was 2.135° C., at 40 seconds 3.05°, at 50 seconds 3.92°, at 60 seconds 4.16°, and the final temperature was 4.200°, the total rise was 2.07°; 60 per cent of it was 1.24°. The temperature to be observed was then 2.14°+1.24°=3.38°. Referring to the observations at 40 and 50 seconds, the temperatures were respectively 3.05 and 3.92°. The time corresponding to the temperature of 3.38° was, therefore:

$$40 + \frac{3.38 - 3.05}{3.92 - 3.05} \times 10 = 44 \text{ seconds.}$$

Example.—

OBSERVATIONS.

Water equivalent = 2550 g.
Weight of charge = 1.0535 g.
Approximate rise of temperature expected = 3.2° C.
60 per cent of approximate rise = 1.9° C.

TIME	THERMOMETER READINGS, DEG. CENT.	CORRECTED TEMPERATURE (or corrected Beckmann thermometer readings)
10–21	15.244	Thermometer corrections taken from the certificate.
22	15.250	
23	15.255	
24	15.261	
25	15.266	
(*a*) 26	15.272	15.276° C.
		Charge fired
(*b*) 27.2	17.2[a]	
(*c*) 31	18.500	18.497° C.
32	18.498	
33	18.497	
34	18.498	
35	18.494	
36	18.493	

[a] The initial temperature is 15.27° C.; 60 per cent of the expected rise is 1.9° C. The reading to observe is then 17.2°.

CALCULATION

$r_1 = 0.028° \div 5 = 0.0056°$ per minute. $b - a = 1.2$ minutes.
The corrected initial temperature is

15.276° + 0.0056° × 12	= 15.283°	
$r_2 = 0.007° \div 5 = 0.0014°$ per minute; $c - b = 3.8$ minutes		
The corrected final temperature is		
18.497° + 0.0014° × 3.8	= 18.502°	
Total rise 18.502° − 15.283°	= 3.219°	
Total calories 2550 × 3.219	=	8209
Titration, etc		−7
Calories from 1.0535 g. sample		8202
Calories per gram	7785	
or British thermal units per pound	14013	

The results obtained by the above method of computation and determination is the total heat of combustion at constant volume, with the water in the products of combustion condensed to liquid at the temperature of the calorimeter, that is, about 20 to 35° C.

Net heat of combustion at 20°, shall refer to results corrected for latent heat of vaporization, as follows:

Total heat of combustion in B. t. u. − 1040 (total hydrogen × 9) = net heat of combustion in B. t. u. per pound.

Also:

Total heat of combustion in calories − 580 (total hydrogen × 9) = net heat of combustion in calories per gram.

NOTES

For anthracite, coke, and coal of high ash content, which do not readily burn completely, the following procedure is recommended:

The inside of the crucible is lined completely with ignited asbestos in a thin layer pressed well down into the angles. The sample is then sprinkled evenly over the surface of the asbestos. Otherwise the procedure is as previously described.

The method of computing the "cooling correction" described in U. S. Bureau of Mines, *Technical Paper No. 8*, pp. 24–28 (1926), may also be used.

TOLERANCES

The permissible differences between two or more determinations shall not exceed the values as specified.

	Permissible Differences	
	Same Laboratory	Different Laboratories
A. On Laboratory Samples, Crushed to Pass through an 840-Micron (No. 20) Sieve.		
Moisture:		
Under 5 per cent	0.2	0.3
Over 5 "	0.3	0.5
B. On Laboratory Samples, Crushed to Pass through a 250-Micron (No. 60) Sieve.		
1. Moisture:		
Under 5 per cent	0.2	0.3
Over 5 "	0.3	0.5
2. Ash:		
No carbonates present	0.2	0.3
Carbonates present	0.3	0.5
Coals with more than 12 per cent of ash, containing carbonate and pyrite	0.5	1.0
3. Volatile Matter:		
Bituminous coals	0.5	1.0
Lignites	1.0	2.0
Cokes	0.2	0.4
4. Sulfur:		
Coal, Under 2 per cent	0.05	0.10
Coal, Over 2 "	0.10	0.20
Coke	0.03	0.05
5. Ultimate Analysis:		
Carbon	0.3	....
Hydrogen	0.07	....
Nitrogen	0.05	....
6. Calorimetric Determination:		
Permissible differences in per cent	0.3	0.5
7. Fusibility of Coal Ash:		
Permissible differences in deg. Cent	30	50

STANDARD METHOD OF SHATTER TEST FOR COKE[1]

Serial Designation: D 141 – 23

This method is issued under the fixed designation D 141; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED IN AMENDED FORM, 1923.

APPARATUS

Shatter Test Machine.

1. The shatter test machine shall consist of a box 18 in. in width, 28 in. in length, and approximately 15 in. in depth, supported above a rigidly mounted cast-iron or steel plate, not less than ½ in. in thickness, 38 in. in width, and 48 in. in length. The inside of the bottom of the box shall be 6 ft. above the plate. The bottom of the box shall consist of two doors hinged lengthwise and latched so that they will swing open freely and not impede the fall of the coke. Boards about 8 in. in height should be placed around the plate so that no coke is lost. To prevent the breakage of coke, which may occur while placing the sample in the box, the box shall be constructed so that it can be lowered to a convenient level, which is best done by means of a pulley and counter-weight. A convenient form of shatter test machine is shown in Fig. 1.

Screens.

2. For determining the breakage of coke, square-mesh screens with the following openings shall be used: 2 in., 1½ in., 1 in., and ½ in. A circular screen, 2 ft. in diameter, is satisfactory.

Unit of Sampling.

3. Each carload, or its equivalent, shall be considered a unit for sampling.

SAMPLING

Place of Sampling.

4. (*a*) If the shatter test is to be used to indicate the probable breakage of coke on handling, the sample should be taken before the coke is subjected to possible breakage resulting from screening and loading into cars. In the case of by-product coke, the sample should be collected from the coke wharf; while samples of beehive coke should be collected as it is delivered from the ovens.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-5 on Coal and Coke.

(*b*) If the shatter test is to be used to indicate the fitness of the coke for furnace or cupola use, the sample should be collected at the point of delivery.

Sampling at Source.

5. (*a*) *By-Product Coke.*—About 75 lb. of representative pieces of coke, none of which would in any position pass through a 2-in. square-mesh screen, shall be selected from the coke wharf for each test. This is best accomplished by dividing the coke on the wharf into approximately equal areas, and selecting an equal number of pieces from each

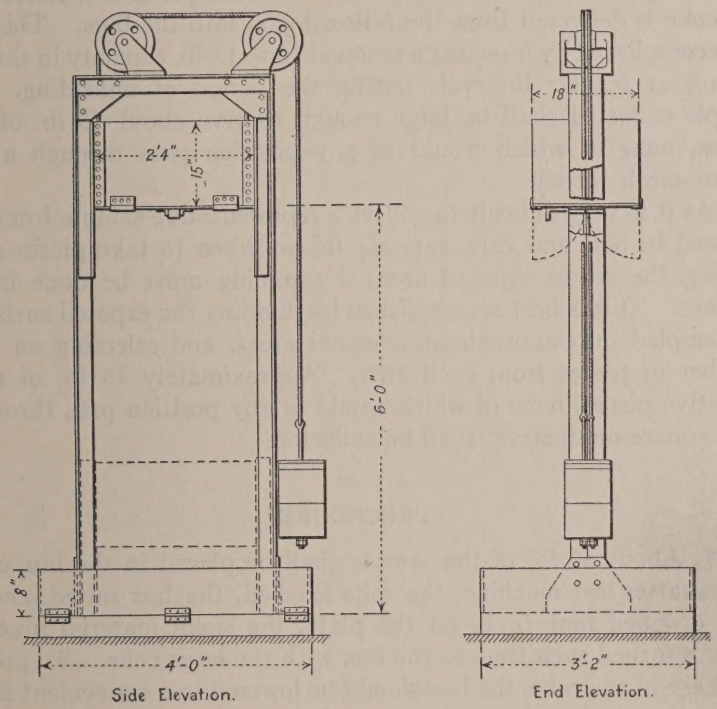

FIG. 1.—Shatter Test Machine.

area. Each piece selected shall be approximately equal in length to one-half the width of the coke-ovens, and should show a "cauliflower" end produced at the walls of the ovens, and an "inner" end produced at the center of the ovens.

(*b*) *Beehive Coke.*—About 75 lb. of representative pieces of coke shall be selected for each test as the coke is drawn from the ovens. This is best done by selecting full-length pieces, or their equivalent, as the

coke is being drawn from previously determined points in the oven, so that they include pieces from the front, sides, center, and back. If the individual pieces as taken from the ovens are too large and bulky, the 75-lb. sample shall be collected by removing three small pieces from each large piece—one from the top, the middle, and the bottom. The sample finally collected should be composed of an equal number of pieces showing top, middle, and bottom. None of the pieces comprising the sample shall in any position pass through the 2-in. square-mesh screen.

Sampling at Delivery.

6. *By-Product and Beehive Coke.*—The sample is best collected as the coke is delivered from the railroad cars into the bins. This may be accomplished by inserting a scoop of 10 to 15-lb. capacity in the coke stream at regular intervals during the period of unloading. The sample collected shall be large enough to give about 75 lb. of coke pieces, none of which would in any position pass through a 2-in. square-mesh screen.

As it is very difficult to collect a representative sample from coke exposed in bins and cars, care should be taken to take pieces representing the entire exposed area, if sampling must be done in this manner. This is best accomplished by dividing the exposed surface to be sampled into approximately equal areas, and selecting an equal number of pieces from each area. Approximately 75 lb. of representative pieces, none of which would in any position pass through a 2-in. square-mesh sieve, shall be collected.

PROCEDURE

Procedure.

7. About 50 lb. of the sample shall be placed in the box of the coke shatter test machine, the coke levelled, the box raised, and the coke dropped four times on the plate, the small material produced being returned each time to the box with the large coke. To prevent breakage of the coke, the box should be lowered to a convenient height when transferring the sample into it. After the fourth drop, the material shall be successively run through the screens mentioned below. The coke should be screened in such increments as will allow all pieces to be in direct contact with the screen openings.

In screening, care should be taken to prevent breakage of the coke pieces. The screen shall be shaken gently until all of the pieces are in direct contact with the meshes. The coke remaining on each screen, and that which passes through, shall be weighed separately. If the sum of these weights shows a loss of over 1 per cent, the test shall be rejected and another made.

The results of the shatter test shall be reported as follows:

Retained on 2-in. screen..... ..... per cent
Passing 2-in. screen, " " 1½-in. " .... per cent
" 1½-in. " , " " 1-in. " .... per cent
" 1-in. " , " " ½-in. " .... per cent
" ½-in. " .... per cent

Number of Tests.

8. Since the average probable error of a single shatter test determination is approximately 2 per cent, it is advisable to make several tests and report the average result.

STANDARD METHOD OF TEST
FOR
VOLUME OF CELL SPACE OF LUMP COKE[1]

Serial Designation: D 167 - 24

This method is issued under the fixed designation D 167; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED, 1924.

Volume of Cell Space.

1. The percentage by volume of cell space of lump coke shall be calculated from the apparent specific gravity of the moisture-free lump coke and the true specific gravity of moisture-free coke passing a 74-micron (No. 200) sieve, as follows:

$$\text{Percentage of cell space} = 100 - 100\,\frac{\text{Apparent specific gravity}}{\text{True specific gravity}}$$

A. Determination of Apparent Specific Gravity

APPARATUS

Apparatus.

2. The apparatus for the determination of the apparent specific gravity of coke shall consist of the following:

(*a*) A suitable container not smaller than approximately 13 in. in height, 22 in. in length and 11 in. in width, or of equivalent size, provided with a spout consisting of a short $\frac{1}{2}$-in. nipple extending horizontally from the container about $2\frac{1}{2}$ in. below the top.

NOTE.—A wash boiler of suitable size to which a spout has been soldered answers the above description.

(*b*) A wire cage or basket of about $\frac{1}{2}$-in. square-mesh screen wire cloth provided with a cover and two long handles, suitable for holding the entire sample of coke and so made as to fit inside the container below the spout.

(*c*) A 3-gal. bucket or other vessel suitable for receiving the displaced water.

(*d*) A pan about 15 in. square and 3 in. in height or the equivalent for containing the coke during the determination of its weight.

(*e*) A scale sensitive to $\frac{1}{2}$ oz.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-5 on Coal and Coke.

SAMPLING

3. Each carload, or its equivalent, shall be considered a unit for sampling. **Unit of Sampling.**

4. (*a*) If the porosity test is desired on run-of-oven coke, the sample should be collected from the coke wharf in the case of by-product coke, while samples of beehive coke should be collected as it is delivered from the ovens. **Place of Sampling.**

(*b*) If the porosity test is desired on coke for furnace or cupola use, the sample should be representative of the material in question and collected at the point of delivery.

5. (*a*) *By-Product Coke.*—About 50 lb. of representative pieces of coke shall be selected from the coke wharf for each test. This is best accomplished by dividing the coke on the wharf into approximately equal areas, and selecting an equal number of pieces from each area. Each piece selected shall be approximately equal in length to one-half of the width of the coke-ovens, and shall show a "cauliflower" end produced at the walls of the ovens, and an "inner" end produced at the center of the ovens. **Sampling at Source.**

(*b*) *Beehive Coke.*—About 50 lb. of representative pieces of coke shall be selected for each test as the coke is drawn from the ovens. This is best done by selecting full-length pieces, or their equivalent, as the coke is being drawn from previously determined points in the oven, so that they include pieces from the front, sides, center, and back. If the individual pieces as taken from the ovens are too large and bulky, the 50-lb. sample shall be collected by removing three small pieces from each large piece—one from the top, the middle, and the bottom. The sample finally collected should be composed of an equal number of pieces showing top, middle and bottom.

6. *By-Product and Beehive Coke.*—The sample is best collected as the coke is delivered from the railroad cars into the bins. This may be accomplished by inserting a scoop of 10 to 15-lb. capacity in the coke stream at regular intervals during the period of unloading. The sample collected shall be large enough to give about 50 lb. of coke pieces, none of which would in any position pass through a 1-in. square-mesh screen. **Sampling at Delivery.**

NOTE.—Since the cage or basket is of ½-in. square-mesh screen wire cloth, it is necessary to have coke pieces that will remain in the cage when the cage is removed from the water.

As it is very difficult to collect a representative sample from coke exposed in bins and cars, care should be taken to take pieces representing the entire exposed area, if sampling must be done in this

manner. This is best accomplished by dividing the exposed surface to be sampled into approximately equal areas, and selecting an equal number of pieces from each area. A 50-lb. sample of representative pieces shall be collected, none of which should in any position pass through a 1-in. square-mesh screen.

PROCEDURE

Procedure.

7. About 25 lb. of coke from the sample shall be selected so as to be representative of the material under consideration with regard to size, shape and general appearance. The coke shall be dried to constant weight at a temperature of from 105 to 200° C. The coke shall be weighed when cool, after shaking and brushing off any adhering dust.

A cork shall be placed in the spout of the container, which has been placed on a level and rigid base or floor. After the empty cage has been placed into the container, water at room temperature shall be poured into the container until the water level is above the spout. After the water has come to rest, the cork shall be removed from the spout and the excess water be permitted to drain out for one minute after the overflow stream starts to discharge drop by drop. The cork shall then be replaced and the cage removed from the water, care being taken to shake all adhering water back into the container. The weighed dried coke sample shall then be placed into the cage and after fastening the cover, the cage containing the coke shall be lowered into the water.

NOTE.—If there is not sufficient capacity in the container above the spout to retain the displaced water, some of the water may be drawn off into a weighed bucket, or other suitable vessel, by removing the cork from the spout while the coke is being lowered.

The cage shall be permitted to remain in the water for 15 minutes with occasional shaking to detach any air bubbles adhering to the surface of the coke, care being taken not to disturb the position of the container. At the end of the 15-minute period, during which the coke shall have been completely submerged at all times, after the water has come to rest, the cork shall be removed and the displaced water permitted to drain into a weighed bucket or other suitable vessel for 1 minute after the overflow stream starts to discharge drop by drop. The cork shall then be replaced, the cage containing the coke removed from the water and permitted to drain for 1 minute. The wet coke shall then be removed from the cage and weighed.

The weight of the displaced water, which has been caught in the bucket, shall then be determined.

8. The apparent specific gravity shall be calculated from the formula:

$$\text{Apparent specific gravity} = \frac{A}{B + (C - A)}$$

in which A = Weight of dry coke;
B = Weight of water displaced by wet coke; and
C = Weight of wet coke.

B. Determination of True Specific Gravity

APPARATUS

9. The apparatus for the determination of the true specific gravity of coke passing a 74-micron (No. 200) sieve shall consist of a Hogarth's specific gravity bottle with side tubulure, having a capacity of about 100 cc. The bottle shall be accurately calibrated so that a table may be constructed giving the contents of the bottle at the room temperatures likely to occur in the laboratory. Apparatus.

NOTE.—This may be done conveniently from data in tables of corrections for determining the true capacities of glass vessels from the weight of water in air, as given in U. S. Bureau of Standards *Circular No. 19*, pp. 52 to 56 (1916).

PROCEDURE

10. A 10-g. portion of 200-mesh coke, which has been previously dried for 1 hour at 105° C., shall be carefully introduced into the specific gravity bottle with a sufficient quantity of distilled water to fill the bottle about one-half full. The bottle shall then be placed on a hot plate and the contents kept boiling for 1 hour, the specific gravity bottle being shaken frequently so as to wash down any coke adhering to the sides. After boiling for 1 hour, the bottle shall be removed from the plate, filled to the tubulure with recently boiled and cooled distilled water, and the stopper inserted. The bottle shall be permitted to stand until the contents have cooled to room temperature. Procedure.

NOTE.—Cooling may be hastened by placing the bottle in water.

After the contents of the bottle have cooled to room temperature, the bottle shall be filled to slightly above the mark on the capillary of the stopper with recently boiled distilled water which has been cooled to room temperature.

NOTE.—This is conveniently done by inserting the end of the tubulure in a small beaker of the distilled water and applying a slight suction on the stopper.

The water level shall be adjusted to the mark on the capillary by touching a piece of filter paper to the end of the tubulure. The bottle shall then be wiped dry and weighed immediately. Immediately after the weighing, the stopper shall be removed and the temperature of the contents taken.

Specific Gravity.

11. The true specific gravity shall be calculated from the formula:

$$\text{True specific gravity} = \frac{W}{W - (W' - P)}$$

in which W = Weight in grams of dry coke;

W' = Weight in grams of bottle + dry coke + water required to fill the bottle;

P = Weight in grams of bottle + water required to fill the bottle.

Duplicate Determinations.

12. The differences in duplicate determinations shall not be more than the following:

Same analyst	0.01
Different analysts	0.02

STANDARD DEFINITIONS OF TERMS RELATING TO COAL AND COKE[1]

Serial Designation: D 121 – 27

These definitions are issued under the fixed designation D 121; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1927.

Proximate Analysis.—In the case of coal and coke, the determination, by prescribed methods, of moisture, volatile matter, fixed carbon (by difference), and ash.

NOTE.—Unless otherwise specified, the term "proximate analysis" does not include determinations of sulfur or phosphorus or any determinations other than those named.

Ultimate Analysis.—In the case of coal and coke, the determination of carbon and hydrogen in the material, as found in the gaseous products of its complete combustion, the determination of sulfur, nitrogen, and ash in the material as a whole, and the estimation of oxygen by difference.

NOTE 1.—The determination of phosphorus is not by definition a part of the ultimate analysis of coal or coke, but may be specified when desired.

NOTE 2.—When the analysis is made on an undried sample, part of the hydrogen and oxygen as determined is present in the free moisture accompanying the coal. Therefore, in comparing coals on the basis of their ultimate analysis, it is advisable always to state the analysis on both the "as-received" and "dry" bases.

NOTE 3.—Inasmuch as some coals contain mineral carbonates, and practically all contain clay or shale containing combined water, a part of the carbon, hydrogen, and oxygen found in the products of combustion may arise from these mineral components.

Moisture.—Essentially water, quantitatively determined by definite prescribed methods which may vary according to the nature of the material.

NOTE 1.—Such methods may not determine all of the water present.

NOTE 2.—In the case of coal and coke the methods employed shall be those prescribed in the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (Serial Designation: D 271) of the American Society for Testing Materials.[2]

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[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee D-5 on Coal and Coke.

[2] See p. 535.

Ash.—Inorganic residue remaining after ignition of combustible substances, determined by definite prescribed methods.

NOTE 1.—Ash may not be identical, in composition or quantity, with the inorganic substances present in the material before ignition.

NOTE 2.—In the case of coal and coke the methods employed shall be those prescribed in the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (Serial Designation: D 271) of the American Society for Testing Materials.[1]

Volatile Matter.—Those products, exclusive of moisture, given off by a material as gas or vapor, determined by definite prescribed methods which may vary according to the nature of the material.

NOTE.—In the case of coal and coke the methods employed shall be those prescribed in the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (Serial Designation: D 271) of the American Society for Testing Materials.[1]

Fixed Carbon.—In the case of coal, coke, and bituminous materials, the solid residue other than ash, obtained by destructive distillation, determined by definite prescribed methods.

NOTE 1.—It is made up principally of carbon, but may contain appreciable amounts of sulfur, hydrogen, nitrogen, and oxygen.

NOTE 2.—In the case of coal and coke the methods employed shall be those prescribed in the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (Serial Designation: D 271) of the American Society for Testing Materials.[1]

[1] See p. 535.

STANDARD SPECIFICATIONS
FOR
STRUCTURAL WOOD JOIST, PLANKS, BEAMS, STRINGERS AND POSTS[1]

Serial Designation: D 245 – 27

These specifications are issued under the fixed designation D 245; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1926; ADOPTED IN AMENDED FORM, 1927.

A. STRUCTURAL GRADES OF LUMBER AND TIMBER AND THE METHOD OF THEIR DERIVATION

Purpose.

1. The purpose of structural grades is to offer means for selecting structural material for strength, and for uniformity in strength, in order that appropriate working stresses may be assigned for its use.

Factors of Strength.

2. The most important factors which influence the strength of structural material are the size, number and location of defects, and the extent of exposure to moisture during use. These factors must all be considered in design or grading if the maximum utilization is to be obtained from the material used.

Structural Grades and Strength.

3. Structural grades control defects by limiting their size and location in accordance with their effect upon strength. Working stresses for each species are recommended by the Forest Products Laboratory, U. S. Forest Service, and take into consideration the allowable defects, the moisture content as determined by conditions of use, and, in the case of southern pine and Douglas fir, rate of growth and percentage of summerwood.

Moisture.

4. Moisture affects the strength of structural timbers both directly and indirectly. The direct effect of loss of moisture is the stiffening and strengthening of the wood fibers. This increase in strength, however, is accompanied by checking, splitting, warping and twisting; as a consequence, some of the strength due to drying is lost. Timbers are also subject, during use, to varying conditions of moisture, from the dry location of a heated building, to the continually wet condition of some pier and dock timbers. All of these conditions are taken into account in recommending working stresses under different conditions of use.

Size Effects.

5. In dimension material, 4 in. and less in thickness, the development of defects during seasoning does not offset the increase in strength from drying as much as in larger sizes, and in these sizes used in dry locations, higher working stresses in extreme fiber in bending can be recommended than in pieces of larger size having proportionately equivalent defects.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

Defects.

6. The principal defects which must be limited in structural grades are: knots, shakes and checks, and angle of grain.

Influence of Knots.

7. The influence of a knot is determined by its location in a piece and the area of its projection on the cross-section of the piece, the method of measurement being such as to give the best approximation of this influence. Knots in posts and heavy beams, which are likely to show only on one face or to run diagonally through the piece, reduce the strength in practically direct proportion to their size as measured. In dimension sizes, such as joist, in which the knot is likely to run directly through the piece, the strength is measured by the square of the effective depth, assuming the knot in its worst position, near the edge of the piece, and the reduction in strength due to the knot is approximately twice the ratio of the size of the knot to the width of the face. In similar material used flat, as plank, the influence of a knot is directly proportional to the size, as on the top and bottom edges of beams.

Location of Knots, Joist and Plank.

8. Knot limitations on edges of wide faces of dimension sizes, for use as joist, are more severe than would be required for use flat, as plank, the sizes applying along the center lines of the wide faces as joist being those which could theoretically apply at any point across the width if used only as plank. It has been found, however, that under practically all conditions of use, knots along the edges of planks are more objectionable than knots along the center lines and this is recognized in some commercial yard grades of plank in a stricter limitation of knots along the edges of the wide faces than along the center lines. The same knot limitations are applied, therefore, to material to be used either as joist or plank, and the same working stresses are assigned for use either on edge or flat.

Increase in Size of Knots.

9. In both joist and beams, knots reduce strength most along the top and bottom edges through the center portion. The sizes of knots permitted in various portions of a joist or beam are limited in accordance with the stresses, and they are allowed to increase toward the ends and toward the center lines of the vertical faces, no knot, however, being permitted of more than double the size allowed at the point of maximum stress.

Small and Large Knots.

10. There is greater proportional distortion of grain around a large knot than around a smaller one, and shrinkage in seasoning causes greater internal stresses, so knot sizes are increased proportionately to width of faces only up to 6-in. top and bottom faces of beams, 12-in. vertical faces of beams and 12-in. faces of dimension material and posts. Beyond these widths of face, increase is proportional to the square root of the ratio of the wider faces to these widths. The distribution and aggregate diameter of knots is limited, as well as the maximum size of the single knot.

Aggregate Diameter of Knots.

11. The aggregate diameter of all knots in the center half of the length on any face of a beam or stringer shall be limited, in direct proportion to an aggregate diameter of twice the width of the face in a grade having 50 per cent of the strength of clear wood. In joist and plank, in dry locations, the aggregate diameter of knots in the center half of the length of any face may be greater by one-half the width of the face than the aggregate diameter permitted in beams and stringers. In posts and columns, the aggregate diameter of all knots in any 6 in. of length, in any grade, shall not exceed twice the size of the maximum knot allowable.

Knots in Joist and Plank.

12. In joist and plank, the mean or average diameter of a knot is taken as its size. In such thin and relatively wide material, whether used on edge or flat, this is a safe measure of the influence of knots on strength, and has the

commercial advantage of being directly applicable to yard grades of lumber. This method of measurement will exclude damaging spike knots, and can be applied to them as well as to round or oval knots.

Knots in Beams.

13. On the top or bottom of a beam, the influence of knots is measured largely by the surface fibers cut. The projection of the knot on a line at a right angle to the edge is, therefore, used. On the vertical face of a beam, the depth to which a knot penetrates is of great importance, while the influence of the number of surface fibers cut, and the amount of grain distortion, is considerably less important than on the horizontal faces. The smallest diameter of the knot is, therefore, used.

Spike Knots.

14. One of the best examples illustrating the reason for the smallest diameter being taken on the vertical face is the splitting of a boxed-heart timber into two pieces. The long spike knots which might be opened up in this way would be no more injurious to the strength of the two pieces than they would as a single knot in a boxed-heart piece, and the two pieces so cut would be less subject to seasoning checks than a boxed-heart piece.

Knots in Columns.

15. In columns there are two factors: area of cross-section occupied by a knot, which would probably be measured best by the small diameter of the knot, and the influence of bending stresses when the column begins to fail, probably measured best by the projection of the knot. In short columns, the area of the cross-section is of primary importance; as the column gets longer, the factor of bending strength increases in importance until the condition of the Euler formula is reached, when stiffness, on which knots have practically no influence, becomes the ruling factor. The average diameter, therefore, is used as that which applies best to the average condition.

Knots and Holes.

16. In grades for structural uses, no distinction is made between intergrown knots and encased knots or knotholes, observation at the Forest Products Laboratory in recent tests having shown that intergrown knots reduce strength fully as much as encased knots or knotholes.

Shakes and Checks.

17. Shakes reduce the area of a beam acting in resistance to shear, and the limitations placed on shakes are based on this reduction. Checks are limited on the same basis as shakes, and no combination of shakes and checks is permitted which would reduce strength to a greater extent than would the allowable size of either separately.

18. Shakes and checks in Dense Select and Select joist and plank shall not exceed when green one-fourth the width of end nor when seasoned one-third the width of end.

Shakes and checks in Common joist and plank shall not exceed when green four-tenths the width of end nor when seasoned four-ninths the width of end.

Shakes and checks in Dense Select and Select beams and stringers shall not exceed when green one-fourth the width of end nor when seasoned one-third the width of end.

Shakes and checks in Common beams and stringers shall not exceed when green four-tenths the width of end nor when seasoned four-ninths the width of end.

Shakes and checks in Dense Select and Select posts and timbers shall not exceed when green four-tenths the width of end nor when seasoned one-half the width of end.

Shakes and checks in Common Posts and Timbers shall not exceed when green one-half the width of end nor when seasoned six-tenths the width of end.

Slope of Grain.

19. Slope of grain, resulting either from diagonal sawing or from spiral or twisted grain in the log, is limited in accordance with the recommendation of the Forest Products Laboratory, based on the results of detailed study of the effect of cross and spiral grain upon strength, and the weakening of material by checks which invariably develop and, without exception, follow the grain. There is not much reduction in strength from cross grain until an angle of grain of 1 in 40 is reached. From that slope in a beam an angle of grain of 1 in 20 reduces the strength about one-eight; 1 in 15, about one-quarter; 1 in 11, about three-eights; and 1 in 8, about one-half. In a post or column an angle of grain of 1 in 15 reduces the strength about one-eight; 1 in 11, about one-quarter; 1 in 8, about three-eights; and 1 in 6, about one-half.

Wane and Knots.

20. Wane is limited by such consideration as bearing area, nailing edge, appearance, etc., rather than by effect on strength. The percentage reduction in strength resulting from wane toward the center of a beam is about double the percentage reduction in cross-sectional area. No combination of wane and knots is permitted which would reduce the strength more than the maximum allowable knot. The occurrence of maximum wane and maximum knot in the same cross-section at the center of a beam would be so rare, however, and the effect of allowable maximum wane is so small a percentage of the effect of maximum allowable knot, that the additional reduction in strength beyond the effect of the knot would be slight and it is usually unnecessary to give attention to combination of wane and knot.

Pitch Pockets.

21. Pitch pockets are ordinarily not defects in a structural grade. A large number, however, indicates a general lack of bond, and such a piece should be carefully inspected for shakes.

Heartwood and Sapwood.

22. Heartwood and sapwood have been found by the Forest Products Laboratory to be of equal strength, and no requirement of heartwood need be made when strength alone is the governing factor. Heart requirement, when durability of untreated material under exposure is a factor, as in bridges, trestles, docks and piers, or in damp buildings, or buildings in which conditions of high humidity prevail, may be specified in any grade, according to exposure and use. When preservative treatment is to be applied, there should be no restriction as to sapwood, as sapwood is easier to treat than heartwood and a large amount is to be preferred.

Density and Strength.

23. The density of the wood substance of all species is practically the same. The dry weight is, therefore, a measure of the amount of wood substance present; and on the amount of wood substance present depends the strength of the clear wood. No pieces of exceptionally light weight are permitted in the Select grades, but light-weight pieces otherwise of Select grade may be accepted in the Common grades.

Density and Summerwood.

24. In southern pine and Douglas fir, the proportion of summerwood, the dark portion of the annual ring, furnishes a practical means of estimating density. Selection of these species for density, to the extent that dense material is commercially available, assures material of the highest character from the standpoint of strength, and uniformity in strength, in the clear wood.

Rate of Growth and Strength.

25. Selection of these species for rate of growth is not as great an assurance of increased strength as selection for percentage of summerwood, but for many purposes selection for rate of growth will assure material of suitable type. Close grain, that is, not less than six nor more than twenty annual rings per inch, is required in the Select Structural grades of these species.

Contrast Between Summerwood and Springwood.

26. In acceptance for density the contrast in color between summerwood and springwood should be distinct. Absence of contrast occasionally occurs in bands of growth rings which appear on the whole darker in color than the adjacent material. The summerwood merges into the springwood abnormally with a gradual change of color, leaving practically no material which has the normal appearance of springwood. Such material has been called by a number of names, including proud wood, red wood, and compression wood. It has a decided end shrinkage, is weak in tension, and even a small part of a cross-section of this character is undesirable in high-class structural timbers.

Minimum Requirements and Maximum Defects.

27. Structural grades specify minimum requirements and maximum defects, all of which may be present at one time. When a particular piece which is being inspected, therefore, is slightly below the provisions of the grade in some respects but is of average density or above, the relative effect on the properties affected should be given consideration.

Re-inspection.

28. In inspection for density, reasonable variation of opinion between inspectors should be recognized. A fair provision for re-inspection of a particular lot of timbers for density would be that for every three timbers accepted as having one-third or more summerwood, one of the remaining timbers be accepted if having between 30 and 33⅓ per cent summerwood.

Yard Grades and Cutting Grades.

29. A large percentage of material in standard yard grades of "dimension" and "timbers" will meet the additional requirements of structural grades for joist and plank, and posts and timbers, and material to meet these requirements can easily be selected from local stocks: Select from Select Common and Merchantable grades, and Common from No. 1 Common grade. Beams and stringers vary materially in size and are not stocked extensively. These are essentially special order grades.

Joist and Plank, Beams and Stringers.

30. As previously noted the provisions of the joist and plank grades are such that material graded by them may be used on edge, as joist or rafters, or flat, as scaffold plank or factory flooring. Joist and plank grades apply to material not thicker than 4 in. Material thicker than 4 in. for use in bending should be graded by beam and stringer grades. In such material with loads applied to the wide face, the knot requirements for this face are those for the narrow face as given in the rules.

Timbers Used as Beams and Stringers.

31. Material to be used for such purposes as caps, bridge ties, etc., where strength in bending is a factor, should be specified in beam and stringer grades although of shape more commonly considered as of timber grades, as the method of measuring knots in post and timber grades makes it impracticable to assign bending stresses to them. Caps and bridge ties are often square or have horizontal faces wider than the vertical faces, in contrast to beams and stringers in which the narrow faces are horizontal faces and the wide faces are vertical, and this should be noted in applying the knot provisions of the beam and stringer grade to such material.

INTRODUCTION TO STRUCTURAL RULES

32. The following rules for Structural Grades conform to the "Basic Provisions for the Selection and Inspection of Softwood Dimension and Timbers Where Working Stresses are Required" accepted at the General Lumber Conference, Washington, D. C., May 1, 1925, as the basis for the preparation of grading rules for structural material.

33. They are complete rules, covering all conditions necessary for consideration in structural grading, and are divided into sections from which combinations are made covering specific purposes and conditions.

34. These specifications may be used for mill orders, selection from or appraisal of stock on hand in either manufacturers', middlemen's or users' stock.

35. The rules cover the following grades and use classifications:

Grades:

DENSE SELECT.....Douglas Fir and Southern Pine
SELECT.....Douglas Fir and Southern Pine
SELECT.....Other Softwood Species
COMMON.....All Softwood Species

Uses:

JOIST AND PLANK.....Joist, Rafters, Bracing, Scaffold Plank, Factory Flooring, etc.
BEAMS AND STRINGERS..Beams, Girders, Stringers, Bridge Ties, Caps, etc.
POSTS AND TIMBERS.....Posts, Sills, Caps, Timbers, etc.

Optional Provisions:

WANE.....Where Permissible
SQUARE EDGES.....Where Required or Desired
HEARTWOOD REQUIREMENT.....For Durability of Untreated Timbers
SAPWOOD PERMISSIBILITY.....For Material to be Treated

Sizes of Joist and Plank

(Joist, Rafters, Scaffold Plank, Factory Flooring, etc.)

Nominal thickness	2 to 4 in.
Nominal widths	4 in. and wider
Standard thickness	S1S or S2S: $\frac{3}{8}$ in. off
Standard widths	2 to 7 in., S1E or S2E: $\frac{3}{8}$ in. off 8 in. and wider, S1E or S2E: $\frac{1}{2}$ in. off

Sizes of Beams and Stringers

(Beams, Girders, Stringers, etc.)

Nominal thickness.....5 in. and thicker
Nominal widths.....8 in. and wider

Sizes of Posts and Timbers

(Posts, Caps, Sills, Timbers, etc.)

Nominal sizes.....6 by 6 in. and larger

B. SPECIFICATION REQUIREMENTS FOR STRUCTURAL WOOD JOIST, PLANK, BEAMS, STRINGERS, AND POSTS

TIMBER SIZE REQUIREMENTS

Joist and Plank, Surfaced.

1*A*. Structural joist and plank shall be when surfaced S1S or S2S not thinner than the nominal dimension less $\frac{3}{8}$ in. and when surfaced S1E or S2E not narrower than the nominal width less $\frac{3}{8}$ in. for sizes 2 to 7 in., inclusive, and less $\frac{1}{2}$ in. for sizes 8 in. and wider.

1*B*. Structural beams and stringers, and posts and timbers shall be when surfaced S1S, S1E, S2S, S4S not smaller than the nominal size less $\frac{3}{8}$ in. for sizes 7 in. and less, and less $\frac{1}{2}$ in. for sizes 8 in. and over. **Beams and Stringers, Posts and Timbers, Surfaced.**

1*C*. Rough structural joist and plank shall not be thinner than the nominal dimension less $\frac{1}{4}$ in. and not narrower than the nominal width less $\frac{1}{4}$ in. for sizes 2 to 7 in., inclusive, and less $\frac{3}{8}$ in. for sizes 8 in. and wider. **Joist and Plank, Rough.**

1*D*. Rough structural beams and stringers, and posts and timbers shall not be smaller than the nominal size less $\frac{1}{4}$ in. for sizes 7 in. and less, and less $\frac{3}{8}$ in. for sizes 8 in. and over. **Beams and Stringers, Posts and Timbers, Rough.**

GRADE REQUIREMENTS

2. This material shall contain only sound wood. **Sound Wood.**

GENERAL

3. (*a*) No pieces of exceptionally light weight shall be permitted, except that light weight pieces otherwise of Select grade may be accepted in Common grade. **Weight.**

(*b*) Shake shall be measured on the ends of a piece, and its size shall be taken as the shortest distance between lines enclosing the shake and parallel to the wide faces of the piece. Checks and splits shall be limited as provided for shakes. No checks or combinations of checks with shakes which would reduce the strength to a greater extent than the allowable shake shall be permitted. **Shake, Checks and Splits.**

(*c*) Where wane is permitted there shall be no combination of wane and knots which would reduce the strength more than the maximum allowable knot. **Wane and Knots.**

(*d*) Cluster knots and knots in groups are not permitted. **Cluster Knots.**

(*e*) Knot holes and holes from other causes than knots shall be permitted as provided for knots. **Holes.**

(*f*) The size of a knot shall be measured on the section of the knot appearing on the surface under consideration. **Knot Measurement.**

(*g*) When the mean or average diameter of a knot is specified, the size shall be taken as the average of the maximum and minimum diameters. **Mean Diameter.**

(*h*) Knot sizes specified shall be applied to spike knots as well as to round knots. **Spike Knots.**

(*i*) The mean or average diameter of a spike knot shall be taken as the average of its length and its maximum width. **Spike Knot Mean Diameter.**

KNOTS

Joist and Plank

Wide Faces. 4. (*a*) On the wide faces of joist and plank, the measurement of a knot shall be made on the mean or average diameter.

Narrow Faces. (*b*) On the narrow faces of joist and plank, the size of a knot shall be taken as its width between lines parallel to the edges of the piece.

Increase to Ends. (*c*) The size of knots on the narrow faces and edges of wide faces of joist and plank may increase proportionately from the size allowed in the middle third to twice that size at the ends of the pieces.

Increase to Center Line. (*d*) The size of knots on the wide faces of joist and plank may increase proportionately from the size allowed at the edge to that allowed at the center line.

Beams and Stringers

Narrow Faces. 5. (*a*) On the narrow or horizontal faces of beams and stringers the size of a knot shall be taken as its width between lines parallel to the edges of the timber.

Wide Faces. (*b*) On the wide or vertical faces of beams and stringers the smallest diameter of a knot shall be taken as its size.

Edges. (*c*) Knots on the edges of wide or vertical faces of beams and stringers are limited to the same size as on the adjacent narrow or horizontal faces, except that the size is measured on the least diameter of the knot instead of on its width between lines parallel to the edges of the timber.

Increase to Ends. (*d*) The size of knots on the narrow or horizontal faces and edges of wide or vertical faces of beams and stringers may increase proportionately from the size allowed in the middle third to twice that size at the ends of the piece.

Increase to Center Line. (*e*) The size of knots on the wide or vertical faces of beams and stringers may increase proportionately from the size allowed at the edge to that allowed at the center line.

Posts and Timbers

Mean Diameter. 6. In posts and timbers, the measurement of a knot shall be made on the mean or average diameter.

7. *Maximum Size of Knots in Dense Select and Select Joist and Plank.*—

(*a*) KNOTS ON WIDE FACES

WIDTH OF FACE, IN.	SIZE OF KNOT On or Near Edge, Middle Third of Length	Center Line of Face
4	$\frac{3}{4}$ in.	$1\frac{1}{4}$ in.
6	1 "	2 "
8	$1\frac{3}{8}$ "	$2\frac{5}{8}$ "
10	$1\frac{3}{4}$ "	$3\frac{1}{4}$ "
12	$2\frac{1}{8}$ "	4 "
14	$2\frac{3}{8}$ "	$4\frac{1}{4}$ "
16	$2\frac{1}{2}$ "	$4\frac{5}{8}$ "

(*b*) KNOTS ON NARROW FACES OF BOXED-HEART PIECES

THICKNESS OF PIECE, IN.	SIZE OF KNOT Middle Third of Length
2	$\frac{5}{8}$ in.
3	1 "
4	$1\frac{1}{4}$ "

(*c*) The sum of the diameters of all knots within the center half of the length of a joist or plank on any face shall not exceed one and one-half times the width of face on which they occur.

8. *Maximum Size of Knots in Common Joist and Plank.*—

(*a*) KNOTS ON WIDE FACES

WIDTH OF FACE, IN.	SIZE OF KNOT On or Near Edge, Middle Third of Length	Center Line of Face
4	1 in.	$1\frac{3}{4}$ in.
6	$1\frac{1}{2}$ "	$2\frac{1}{2}$ "
8	2 "	$3\frac{3}{8}$ "
10	$2\frac{1}{2}$ "	$4\frac{1}{4}$ "
12	3 "	$5\frac{1}{8}$ "
14	$3\frac{1}{4}$ "	$5\frac{5}{8}$ "
16	$3\frac{3}{8}$ "	6 "

(*b*) KNOTS ON NARROW FACES OF BOXED-HEART PIECES

THICKNESS OF PIECE, IN.	SIZE OF KNOT Middle Third of Length
2	$\frac{7}{8}$ in.
3	$1\frac{1}{4}$ "
4	$1\frac{3}{4}$ "

(*c*) The sum of the diameters of all knots within the center half of the length of a joist or plank on any face shall not exceed two times the width of face on which they occur.

9. (*a*) *Maximum Size of Knots in Dense Select and Select Beams and Stringers.*—

Width of Face, in.	Size of Knot: Narrow or Horizontal Face, Middle Third of Length	Size of Knot: Center Line of Wide or Vertical Face
5	1¼ in.	1¼ in.
6	1½ "	1½ "
8	1¾ "	2 "
10	2 "	2½ "
12	2⅛ "	3 "
14	2¼ "	3¼ "
16	2⅜ "	3⅜ "
18	..	3⅝ "
20	..	3⅞ "
22	..	4 "
24	..	4¼ "

(*b*) The sum of the diameters of all knots within the center half of the length of a beam or stringer on any face shall not exceed the width of face on which they occur.

10. (*a*) *Maximum Size of Knots in Common Beams and Stringers.*—

Width of Face, in.	Size of Knot: Narrow or Horizontal Face Middle Third of Length	Size of Knot: Center Line of Wide Face or Vertical Face
5	2 in.	2 in.
6	2⅜ "	2⅜ "
8	2¾ "	3⅛ "
10	3⅛ "	4 "
12	3⅜ "	4¾ "
14	3⅝ "	5⅛ "
16	3⅞ "	5½ "
18	..	5⅞ "
20	..	6⅛ "
22	..	6½ "
24	..	6¾ "

(*b*) The sum of the diameters of all knots within the center half of the length of a beam or stringer on any face shall not exceed one and one-half times the width of face on which they occur.

11. (*a*) *Maximum Size of Knots in Dense Select and Select Posts and Timbers.*—

Width of Face, in.	Size of Knot
6	1½ in.
8	2 "
10	2½ "

WIDTH OF FACE, IN.	SIZE OF KNOT
12	3 in.
14	3½ "
16	3⅜ "
18	3⅝ "
20	3⅞ "
22	4 "
24	4¼ "

(*b*) The sum of the diameters of all knots in any 6 in. of length of a post or timber shall not exceed twice the size of the maximum knot allowable.

12. (*a*) *Maximum Size of Knots in Common Posts and Timbers.*—

WIDTH OF FACE, IN.	SIZE OF KNOT
6	2⅜ in.
8	3⅛ "
10	4 "
12	4¾ "
14	5⅛ "
16	5½ "
18	5⅞ "
20	6⅛ "
22	6½ "
24	6¾ "

(*b*) The sum of the diameters of all knots in any 6 in. of length of a post or timber shall not exceed twice the size of the maximum knot allowable.

SHAKE AND CHECKS

13. *Maximum Shake and Checks in Dense Select and Select Joist and Plank.*—

WIDTH OF END, IN.	SIZE OF SHAKE OR CHECK	
	Green	Seasoned
2	½ in.	⅝ in.
3	¾ "	1 "
4	1 "	1¼ "

14. *Maximum Shake and Checks in Common Joist and Plank.*—

WIDTH OF END, IN.	SIZE OF SHAKE OR CHECK	
	Green	Seasoned
2	¾ in.	⅞ in.
3	1⅛ "	1¼ "
4	1½ "	1¾ "

15. *Maximum Shake and Checks in Dense Select and Select Beams and Stringers.—*

Width of End, in.	Size of Shake or Check	
	Green	Seasoned
6	1½ in.	2 in.
8	2 "	2⅝ "
10	2½ "	3¼ "
12	3 "	4 "
14	3½ "	4⅝ "
16	4 "	5¼ "
18	4½ "	6 "
20	5 "	6⅝ "
22	5½ "	7¼ "
24	6 "	8 "

16. *Maximum Shake and Checks in Common Beams and Stringers.—*

Width of End, in.	Size of Shake or Check	
	Green	Seasoned
5	2 in.	2⅛ in.
6	2⅜ "	2⅝ "
8	3⅛ "	3½ "
10	4 "	4⅜ "
12	4¾ "	5¼ "
14	5½ "	6⅛ "
16	6⅜ "	7 "
18	7⅛ "	8 "
20	8 "	8⅞ "
22	8¾ "	9¾ "
24	9½ "	10⅝ "

17. *Maximum Shake and Checks in Dense Select and Select Posts and Timbers.—*

Width of End, in.	Size of Shake or Check	
	Green	Seasoned
5	2 in.	2½ in.
6	2⅜ "	3 "
8	3⅛ "	4 "
10	4 "	5 "
12	4¾ "	6 "
14	5½ "	7 "
16	6⅜ "	8 "
18	7⅛ "	9 "
20	8 "	10 "
22	8¾ "	11 "
24	9½ "	12 "

18. *Maximum Shake and Checks in Common Posts and Timbers.*—

WIDTH OF END, IN.	SIZE OF SHAKE OR CHECK	
	Green	Seasoned
6	3 in.	3½ in.
8	4 "	4¾ "
10	5 "	6 "
12	6 "	7⅛ "
14	7 "	8⅜ "
16	8 "	9½ "
18	9 "	10¾ "
20	10 "	12 "
22	11 "	13⅛ "
24	12 "	14⅜ "

SLOPE OF GRAIN

19*A*. *Dense Select and Select Joist and Plank.*—The slope of grain in the center half of length shall not exceed 1 in 12.

19*B*. *Common Joist and Plank.*—The slope of grain in the center half of length shall not exceed 1 in 10.

19*C*. *Dense Select and Select Beams and Stringers.*—The slope of grain in the center half of length shall not exceed 1 in 15.

19*D*. *Common Beams and Stringers.*—The slope of grain in the center half of length shall not exceed 1 in 10.

19*E*. *Dense Select and Select Posts and Timbers.*—The slope of grain shall not exceed 1 in 10.

19*F*. *Common Posts and Timbers.*—The slope of grain shall not exceed 1 in 8.

WANE OR SQUARE EDGES

20*A*. *Wane, Dense Select and Select Grade.*—Wane is permitted, not exceeding one-eighth the width of any face. **Wane One-Eighth.**

20*B*. *Wane, Common Grade.*—Wane is permitted, not exceeding one-fourth the width of any face. **Wane One-Fourth.**

20*C*. *Square Edges.*—All edges must be square. **Square.**

HEARTWOOD AND SAPWOOD

Durability Untreated

Heartwood requirements shall be specified as required from the following:

Heartwood Provisions.—

21*A*. Joist and plank shall have not less than 85 per cent of heart on the two faces, as measured across the faces anywhere in the length of the piece. **Joist and Plank.**

Beams and Stringers.

21*B*. Beams and stringers shall have not less than 85 per cent heart on each of the four faces measured across the faces anywhere in the length of the piece.

Timbers, 85 per cent.

21*C*. Timbers designated 85-per-cent shall have not less than 85 per cent heart on each of the four faces, measured across the face anywhere in the length of the piece.

Timbers, One Face All Heart, Others 85-per-cent.

21*D*. Timbers designated one face all heart, others 85-per-cent shall have all heart on one narrow face, the other narrow face and the two sides shall have not less than 85 per cent of heart, measured across the face or sides anywhere in the length of the piece.

Timbers, One Face All Heart, Others 75-per-cent.

21*E*. Timbers designated one face all heart, others 75-per-cent shall have all heart on one narrow face, the other narrow face and the two sides shall have not less than 75 per cent of heart measured across the face or sides anywhere in the length of the piece.

For Treatment

Provision for sapwood for timber to be treated is covered by the following:

Sapwood Provisions.—

Sapwood not Restricted.

22. There is no restriction as to sapwood for this material.

Rate of Growth and Density

Select.

Southern pine or Douglas fir of Select grade is to be selected for close grain.

Dense Select.

Southern pine or Douglas fir of Dense Select grade is to be selected for density.

Close Grain

Close Grain.

23. Douglas fir or southern pine shall be of close grain, averaging on either one end or the other not less than six nor more than twenty annual rings per inch measured over a 3-in. portion of a radial line located as described below and representative of the average growth on the cross-section. Pieces averaging from five to six annual rings per inch shall be accepted as the equivalent of close grain if having one-third or more summerwood.

Density

Density.

24. Douglas fir or southern pine shall be dense, averaging on either one end or the other not less than six annual rings per inch and, in addition, one-third or more summerwood measured over a 3-in. portion of a radial line located as described below and representative

of the average growth on the cross-section. The contrast in color between summerwood and springwood shall be distinct. Coarse-grained material excluded by this rule shall be accepted as dense if averaging one-half or more summerwood.

Close Grain or Density

Radial Line Not Representative.

25. When the radial line specified is not representative, it shall be shifted sufficiently to present a fair average, but the distance from the pith to the beginning of the 3-in. portion of the line in boxed heart pieces shall not be changed.

Close Grain on Two Radial Lines.

26. In case of disagreement in the measurement of close grain, two radial lines shall be chosen, and the number of rings shall be the average determined on these lines.

Density on Two Radial Lines.

27. In case of disagreement in the measurement of density, two radial lines shall be chosen, and the number of rings and summerwood shall be the average determined on these lines.

Location of Radial Line

In Douglas Fir.—

Sidecut Pieces.

28. (*a*) In sidecut pieces of Douglas fir, the radial line shall be at a right angle to the annual rings and the center of the 3-in. portion of the line shall be at the center of the end of the piece.

Boxed-Heart Pieces.

(*b*) In boxed-heart pieces, the line shall run from the pith to the corner farthest from the pith. When the least dimension is 6 in. or less, the 3-in. portion of the line shall begin at a distance of one inch from the pith. When the least dimension is more than 6 in., the 3 in. portion of the line shall begin at a distance from the pith equal to 2 in. less than one-half the least dimension of the piece.

(*c*) If a 3-in. portion of the radial line cannot be obtained, the measurement shall be made over as much of the 3-in. portion as is available.

In Southern Pine.—

Boxed-Heart Pieces.

29. (*a*) In boxed-heart pieces of southern pine, the measurement shall be made over the third, fourth and fifth inches from the pith along the radial line.

Pith Not Present.

(*b*) In cases where timbers do not contain the pith, and it is impossible to locate it with any degree of accuracy, the inspection shall be made over 3 in. on an approximate radial line beginning at the edge nearest the pith in timbers over 3 in. in thickness and on the second inch nearest to the pith in timbers 3 in. or less in thickness.

Pith Present, But less than 5-in. Radial Line.

(*c*) In material containing the pith but not a 5-in. radial line, which is less than 2 by 8 in. in section or less than 8 in. in width, that does not show over 16 sq. in. on the cross-section, the inspection shall apply to the second inch from the pith. In larger material that does not show a 5-in. radial line, the inspection shall apply to the 3 in. farthest from the pith.

C. STRUCTURAL GRADES AND REFERENCE CODE

The individual specifications are divided into sections with general numbers for reference.

Where alternate requirements are listed, each has a capital letter added to the general number. For any material, appropriate choice must be made of such paragraphs.

Where several paragraphs occur under the general number and each is designated by a small letter, all such paragraphs must be used in making a complete specification.

In cases where only a small number of sizes are ordered it is permissible to shorten the tables of maximum defects by only copying sizes of defects corresponding with the sizes being ordered.

Three complete specifications are shown to illustrate the application of the code.

These are followed by a complete list of numbered specifications, brief description of material and appropriate code numbers of sections required to write a complete specification for this material.

TYPICAL SPECIFICATIONS FOR DENSE SELECT BEAMS AND STRINGERS (SOUTHERN PINE) WITH SQUARE EDGES AND HEARTWOOD REQUIREMENT FOR USE UNTREATED, ROUGH

SPECIFICATION REFERENCE NO. 1 IN SUB-DIVISION *D* ON CODED SPECIFICATIONS

TIMBER SIZE REQUIREMENTS

1*D*. Rough structural beams and stringers shall not be smaller than the nominal size less $\frac{1}{4}$ in. for sizes 7 in. and less, and less $\frac{3}{8}$ in. for sizes 8 in. and over.

GRADE REQUIREMENTS

2. The material shall contain only sound wood.

GENERAL

3. (*a*) No pieces of exceptionally light weight shall be permitted except that light weight pieces otherwise of Select grade may be accepted in Common grade.

(*b*) Shake shall be measured on the ends of a piece, and its size shall be taken as the shortest distance between lines enclosing the shake and parallel to the wide faces of the pieces. Checks and splits shall be limited as provided

for shakes. No checks or combinations of checks with shakes which would reduce the strength to a greater extent than the allowable shake shall be permitted.

(*c*) Where wane is permitted there shall be no combination of wane and knots which would reduce the strength more than the maximum allowable knot.

(*d*) Cluster knots and knots in groups are not permitted.

(*e*) Knot holes and holes from other causes than knots shall be permitted as provided for knots.

(*f*) The size of a knot shall be measured on the section of the knot appearing on the surface under consideration.

(*g*) When the mean or average diameter of a knot is specified, the size shall be taken as the average of the maximum and minimum diameters.

(*h*) Knot sizes specified shall be applied to spike knots as well as to round knots.

(*i*) The mean or average diameter of a spike knot shall be taken as the average of its length and its maximum width.

KNOTS

Beams and Stringers

5. (*a*) On the narrow or horizontal faces of beams and stringers the size of a knot shall be taken as its width between lines parallel to the edges of the timber.

(*b*) On the wide or vertical faces of beams and stringers the smallest diameter of a knot shall be taken as its size.

(*c*) Knots on the edges of wide or vertical faces of beams and stringers are limited to the same size as on the adjacent narrow or horizontal faces, except that the size is measured on the least diameter of the knot instead of on its width between lines parallel to the edges of the timber.

(*d*) The size of knots on the narrow or horizontal faces and edges of wide or vertical faces of beams and stringers may increase proportionately from the size allowed in the middle third to twice that size at the ends of the piece.

(*e*) The size of knots on the wide or vertical faces of beams and stringers may increase proportionately from the size allowed at the edge to that allowed at the center line.

9. (*a*) *Maximum Size of Knots in Dense Select and Select Beams and Stringers.*—

WIDTH OF FACE, IN.	SIZE OF KNOT	
	Narrow or Horizontal Face, Middle Third of Length	Center Line of Wide or Vertical Face
5	1¼ in.	1¼ in.
6	1½ "	1½ "
8	1¾ "	2 "
10	2 "	2½ "
12	2⅛ "	3 "
14	2¼ "	3¼ "
16	2⅜ "	3⅜ "
18	..	3⅝ "
20	..	3⅞ "
22	..	4 "
24	..	4¼ "

(*b*) The sum of the diameters of all knots within the center half of the length of a beam or stringer on any face shall not exceed the width of the face on which they occur.

SHAKE AND CHECKS

15. *Maximum Shake and Checks in Dense Select and Select Beams and Stringers.*—

WIDTH OF END, IN.	SIZE OF SHAKE OR CHECK	
	Green	Seasoned
6	1½ in.	2 in.
8	2 "	2⅝ "
10	2½ "	3¼ "
12	3 "	4 "
14	3½ "	4⅝ "
16	4 "	5¼ "
18	4½ "	6 "
20	5 "	6⅝ "
22	5½ "	7½ "
24	6 "	8 "

19*C*. The slope of grain in center half of length shall not exceed 1 in 15.

20*C*. All edges must be square.

21*B*. Beams and stringers shall have not less than 85 per cent heart on each of the four faces measured across the faces anywhere in the length of the piece.

24. Southern pine shall be dense, averaging on either one end or the other not less than six annual rings per inch and, in addition, one-third or more summerwood measured over a 3-in. portion of a radial line located as described below and representative of the average growth on the cross-section. The contrast in color between summerwood and spring wood shall be distinct. Coarse-grained material excluded by this rule shall be accepted as dense if averaging one-half or more summerwood.

25. When the radial line specified is not representative, it shall be shifted sufficiently to present a fair average but the distance from the pith to the beginning of the 3-in. portion of the line in boxed-heart pieces shall not be changed.

27. In case of disagreement, two radial lines shall be chosen, and the number of rings and summerwood shall be the average determined on these lines.

29(*a*) In boxed-heart pieces of southern pine, the measurement shall be made over the third, fourth and fifth inches from the pith along the radial line.

(*b*) In cases where the timbers do not contain the pith, and it is impossible to locate it with any degree of accuracy, the inspection shall be made over 3 in. on an approximate radial line beginning at the edge nearest the pith in timbers over 3 in. in thickness and on the second inch nearest to the pith in timbers 3 in. or less in thickness.

(*c*) In material containing the pith but not a 5-in. radial line, which is less than 2 by 8 in. in section or less than 8 in. in width, that does not show over 16 sq. in. on the cross-section, the inspection shall apply to the second inch from the pith. In larger material that does not show a 5-in. radial line, the inspection shall apply to the 3 in. farthest from the pith.

TYPICAL SPECIFICATION FOR SELECT POSTS AND TIMBERS (DOUGLAS FIR) WITH WANE PERMITTED AND SAPWOOD WANTED FOR TREATMENT, SURFACED FOUR SIDES

SPECIFICATION REFERENCE NO. 50 IN SUB-DIVISION *D* ON CODED SPECIFICATIONS

TIMBER SIZE REQUIREMENTS

1*B*. Structural posts and timbers shall be when surfaced S1S, S1E, S2S, S4S, not smaller than the nominal size less $\frac{3}{8}$ in. for sizes 7 in. and less, and less $\frac{1}{2}$ in. for sizes 8 in. and over.

GRADE REQUIREMENTS

2. The material shall contain only sound wood.

GENERAL

3. (*a*) No pieces of exceptionally light weight shall be permitted, except that light weight pieces otherwise of Select grade may be accepted in Common grade.

(*b*) Shake shall be measured on the ends of a piece, and its size shall be taken as the shortest distance between lines enclosing the shake and parallel to the wide faces of the piece. Checks and splits shall be limited as provided for shakes. No checks or combination of checks with shakes which would reduce the strength to a greater extent than the allowable shake shall be permitted.

(*c*) Where wane is permitted there shall be no combination of wane and knots which would reduce the strength more than the maximum allowable knot.

(*d*) Cluster knots and knots in groups are not permitted.

(*e*) Knot holes and holes from other causes than knots shall be permitted as provided for knots.

(*f*) The size of a knot shall be measured on the section of the knot appearing on the surface under consideration.

(*g*) When the mean or average diameter of a knot is specified, the size shall be taken as the average of the maximum and minimum diameters.

(*h*) Knot sizes specified shall be applied to spike knots as well as to round knots.

(*i*) The mean or average diameter of a spike knot shall be taken as the average of its length and its maximum width.

KNOTS

6. In posts and timbers, the measurement of a knot shall be made on the mean or average diameter.

11. (*a*) *Maximum Size of Knots in Dense Select and Select Posts and Timbers.*—

WIDTH OF FACE, IN.	SIZE OF KNOT
6	1½ in.
8	2 "

WIDTH OF FACE, IN.	SIZE OF KNOT
10	$2\frac{1}{2}$ in.
12	3 "
14	$3\frac{1}{4}$ "
16	$3\frac{3}{8}$ "
18	$3\frac{5}{8}$ "
20	$3\frac{7}{8}$ "
22	4 "
24	$4\frac{1}{4}$ "

(*b*) The sum of the diameters of all knots in any 6 in. of length of a post or timber shall not exceed twice the size of the maximum knot allowable.

SHAKE AND CHECKS

17. *Maximum Shake and Checks in Dense Select and Select Posts and Timbers.*—

WIDTH OF END, IN.	SIZE OF SHAKE OR CHECK	
	Green	Seasoned
5	2 in.	$2\frac{1}{2}$ in.
6	$2\frac{3}{8}$ "	3 "
8	$3\frac{1}{8}$ "	4 "
10	4 "	5 "
12	$4\frac{3}{4}$ "	6 "
14	$5\frac{1}{2}$ "	7 "
16	$6\frac{3}{8}$ "	8 "
18	$7\frac{1}{8}$ "	9 "
20	8 "	10 "
22	$8\frac{3}{4}$ "	11 "
24	$9\frac{1}{2}$ "	12 "

19*E*. The slope of grain shall not exceed 1 in 10.

20*A*. Wane is permitted, not exceeding one-eighth the width of any face.

22. There is no restriction as to sapwood for this material.

23. Douglas fir shall be of close grain, averaging on either one end or the other not less than six nor more than twenty annual rings per inch measured over a 3-in. portion of a radial line located as described below and representative of the average growth on the cross-section. Pieces averaging from five to six annual rings per inch shall be accepted as the equivalent of close grain if having one-third or more summerwood.

25. When the radial line specified is not representative, it shall be shifted sufficiently to present a fair average, but the distance from the pith to the beginning of the 3-in. portion of the line in boxed-heart pieces shall not be changed.

26. In case of disagreement, two radial lines shall be chosen, and the number of rings shall be the average determined on these lines.

28(*a*) In side-cut pieces of Douglas fir, the radial line shall be at a right angle to the annual rings and the center of the 3-in. portion of the line shall be at the center of the end of the piece.

(*b*) In boxed-heart pieces the line shall run from the pith to the corner farthest from the pith. When the least dimension is 6 in. or less, the 3-in. portion of the line shall begin at a distance of 1 in. from the pith. When the least dimension is more than 6 in. the 3-in. portion of the line shall begin at a distance from the pith equal to 2 in. less than one-half the least dimension of the piece.

(*c*) If a 3-in. portion of a radial line cannot be obtained, the measurement shall be made over as much of the 3-in. portion as is available.

TYPICAL SPECIFICATION FOR COMMON JOIST AND PLANK WITH WANE PERMISSIBILITY AND NO HEARTWOOD OR SAPWOOD REQUIREMENT, SURFACED S1S, S1E

SPECIFICATION REFERENCE NO. 38 IN SUB-DIVISION *D* ON CODED SPECIFICATIONS

TIMBER SIZE REQUIREMENTS

1*A*. Structural joist and plank shall be, when surfaced S1S or S2S, not thinner than the nominal dimension less $\frac{3}{8}$ in. and when surfaced S1E or S2E not narrower than the nominal width less $\frac{3}{8}$ in. for sizes 2 to 7 in., inclusive, and less $\frac{1}{2}$ in. for sizes 8 in. and wider.

GRADE REQUIREMENTS

2. The material shall contain only sound wood.

GENERAL

3. (*a*) No pieces of exceptionally light weight shall be permitted, except that light-weight pieces otherwise of Select grade may be accepted in Common grade.

(*b*) Shake shall be measured on the ends of a piece, and its size shall be taken as the shortest distance between lines enclosing the shake and parallel to the wide faces of the piece. Checks and splits shall be limited as provided for shakes. No checks or combination of checks with shakes which would reduce the strength to a greater extent than the allowable shake shall be permitted.

(*c*) Where wane is permitted there shall be no combination of wane and knots which would reduce the strength more than the maximum allowable knot.

(*d*) Cluster knots and knots in groups are not permitted.

(*e*) Knot holes and holes from other causes than knots shall be permitted as provided for knots.

(*f*) The size of a knot shall be measured on the section of a knot appearing on the surface under consideration.

(*g*) When the mean or average diameter of a knot is specified, the size shall be taken as the average of the maximum and minimum diameters.

(*h*) Knot sizes specified shall be applied to spike knots as well as to round knots.

(*i*) The mean or average diameter of a spike knot shall be taken as the average of its length and its maximum width.

KNOTS

4. (*a*) On the wide faces of joist and plank, the measurement of a knot shall be made on the mean or average diameter.

(*b*) On the narrow faces of joist and plank, the size of a knot shall be taken as its width between lines parallel to the edges of the piece.

(*c*) The size of knots on the narrow faces and edges of wide faces of joist and plank may increase proportionately from the size allowed in the middle third to twice that size at the ends of the pieces.

(*d*) The size of knots on the wide faces of joist and plank may increase proportionately from the size allowed at the edge to that allowed at the center line.

8. (*a*) *Maximum Size of Knots in Common Joist and Plank, Knots on Wide Faces.*—

WIDTH OF FACE, IN.	SIZE OF KNOT	
	On or Near Edge, Middle Third of Length	Center Line of Face
4	1 in.	$1\frac{3}{4}$ in.
6	$1\frac{1}{2}$ "	$2\frac{1}{2}$ "
8	2 "	$3\frac{3}{8}$ "
10	$2\frac{1}{2}$ "	$4\frac{1}{4}$ "
12	3 "	$5\frac{1}{8}$ "
14	$3\frac{1}{4}$ "	$5\frac{5}{8}$ "
16	$3\frac{3}{8}$ "	6 "

(*b*) *Knots on Narrow Faces of Boxed Heart Pieces.*—

THICKNESS OF PIECE, IN.	SIZE OF KNOT Middle Third of Length
2	$\frac{7}{8}$ in.
3	$1\frac{1}{4}$ "
4	$1\frac{3}{4}$ "

(*c*) The sum of the diameters of all knots within the center half of the length of a joist or plank on any face shall not exceed two times the width of face on which they occur.

SHAKE AND CHECKS

14. *Maximum Shake and Checks in Common Joist and Plank.*—

WIDTH OF END, IN.	SIZE OF SHAKE OR CHECK	
	Green	Seasoned
2	$\frac{3}{4}$ in.	$\frac{7}{8}$ in.
3	$1\frac{1}{8}$ "	$1\frac{1}{4}$ "
4	$1\frac{1}{2}$ "	$1\frac{3}{4}$ "

19*B*. *Common Joist and Plank.*—The slope of grain in the center half of length shall not exceed 1 in 10.

20*B*. *Wane, Common Grade.*—Wane is permitted, not exceeding one-fourth the width of any face.

D. CODED SPECIFICATIONS FOR STRUCTURAL GRADES

INDEX TO CODED SPECIFICATION REFERENCE NUMBERS

GRADES	ADDITIONAL REQUIREMENTS						
	For Use Untreated					For Treating	
	For Durability Heartwood Required			No Heartwood or Sapwood Required		Sapwood not Restricted	
	One Face 100 per cent; Other Three Faces 85 per cent	All Faces 85 per cent	One Face 100 per cent; Other Three Faces 75 per cent				
	Square Edge			Square Edge	Wane Permitted	Square Edge	Wane Permitted
BEAMS AND STRINGERS							
DENSE SELECT: Douglas Fir or Southern Pine.	..	1	..	2	3	4	5
SELECT: Douglas Fir or Southern Pine.	..	6	..	7	8	9	10
SELECT: Any other softwood	..	11	..	12	13	14	15
COMMON: Any softwood.....	..	16	..	17	18	19	20
JOIST AND PLANK							
DENSE SELECT: Douglas Fir or Southern Pine.	..	21	..	22	23	24	25
SELECT: Douglas Fir or Southern Pine.	..	26	..	27	28	29	30
SELECT: Any other softwood	..	31	..	32	33	34	35
COMMON: Any softwood.....	..	36	..	37	38	39	40
POSTS AND TIMBERS							
DENSE SELECT: Douglas Fir or Southern Pine.	61	41	65	42	43	44	45
SELECT: Douglas Fir or Southern Pine.	62	46	66	47	48	49	50
SELECT: Any other softwood	63	51	67	52	53	54	55
COMMON: Any softwood.....	64	56	68	57	58	59	60

Dense Select Beams and Stringers

No. 1. BEAMS AND STRINGERS, DOUGLAS FIR OR SOUTHERN PINE, DENSE SELECT, 85 PER CENT HEARTWOOD, SQUARE EDGES:

1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 5 (*a*+*b*+*c*+*d*+*e*), 9 (*a*+*b*), 15, 19*C*, 20*C*, 21*B*, 24, 25, 27, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 2. BEAMS AND STRINGERS, DOUGLAS FIR OR SOUTHERN PINE, DENSE SELECT, NO HEARTWOOD OR SAPWOOD REQUIREMENT, SQUARE EDGES:

1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 5 (*a*+*b*+*c*+*d*+*e*), 9 (*a*+*b*), 15, 19*C*, 20*C*, 24, 25, 27, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 3. BEAMS AND STRINGERS, DOUGLAS FIR OR SOUTHERN PINE, DENSE SELECT, NO HEARTWOOD OR SAPWOOD REQUIREMENT, WANE PERMITTED:

1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 5 (*a*+*b*+*c*+*d*+*e*), 9 (*a*+*b*), 15, 19*C*, 20*A*, 24, 25, 27, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 4. BEAMS AND STRINGERS, DOUGLAS FIR OR SOUTHERN PINE, DENSE SELECT, SAPWOOD WANTED FOR TREATMENT, SQUARE EDGES:

1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 5 (*a*+*b*+*c*+*d*+*e*), 9 (*a*+*b*), 15,19*C*, 20*C*, 22, 24, 25, 27, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 5. BEAMS AND STRINGERS, DOUGLAS FIR OR SOUTHERN PINE, DENSE SELECT, SAPWOOD WANTED FOR TREATMENT, WANE PERMITTED:

1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 5 (*a*+*b*+*c*+*d*+*e*), 9 (*a*+*b*), 15, 19*C*, 20*A*, 22, 24, 25, 27, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

Select Beams and Stringers

No. 6. BEAMS AND STRINGERS, DOUGLAS FIR OR SOUTHERN PINE, SELECT, 85 PER CENT HEARTWOOD, SQUARE EDGES:

1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 5 (*a*+*b*+*c*+*d*+*e*), 9 (*a*+*b*), 15, 19*C*, 20*C*, 21*B*, 23, 25, 26, 28 (*a*+*b*+*c*), Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 7. BEAMS AND STRINGERS, DOUGLAS FIR OR SOUTHERN PINE, SELECT, NO HEARTWOOD OR SAPWOOD REQUIREMENT, SQUARE EDGES:

1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 5 (*a*+*b*+*c*+*d*+*e*), 9 (*a*+*b*), 15, 19*C*, 20*C*, 23, 25, 26, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 8. BEAMS AND STRINGERS, DOUGLAS FIR OR SOUTHERN PINE, SELECT, NO HEARTWOOD OR SAPWOOD REQUIREMENT, WANE PERMITTED:

1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 5 (*a*+*b*+*c*+*d*+*e*), 9 (*a*+*b*), 15, 19*C*, 20*A*, 23, 25, 26, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 9. BEAMS AND STRINGERS, DOUGLAS FIR OR SOUTHERN PINE, SELECT, SAPWOOD WANTED FOR TREATMENT, SQUARE EDGES:

1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 5 (*a*+*b*+*c*+*d*+*e*), 9 (*a*+*b*), 15, 19*C*, 20*C*, 22, 23, 25, 26, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 10. BEAMS AND STRINGERS, DOUGLAS FIR OR SOUTHERN PINE, SELECT, SAPWOOD WANTED FOR TREATMENT, WANE PERMITTED:

1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 5 (*a*+*b*+*c*+*d*+*e*), 9 (*a*+*b*), 15, 19*C*, 20*A*, 22, 23, 25, 26, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 11. BEAMS AND STRINGERS, SELECT, 85 PER CENT HEARTWOOD, SQUARE EDGES:

1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 5 (*a*+*b*+*c*+*d*+*e*), 9 (*a*+*b*), 15, 19*C*, 20*C*, 21*B*.

No. 12. BEAMS AND STRINGERS, SELECT, NO HEARTWOOD OR SAPWOOD REQUIREMENT, SQUARE EDGES:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 5 (*a*+*b*+*c*+*d*+*e*), 9 (*a*+*b*), 15, 19*C*, 20*C*.

No. 13. BEAMS AND STRINGERS, SELECT, NO HEARTWOOD OR SAPWOOD REQUIREMENT, WANE PERMITTED:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 5 (*a*+*b*+*c*+*d*+*e*), 9 (*a*+*b*), 15, 19*C*, 20*A*.

No. 14. BEAMS AND STRINGERS, SELECT, SAPWOOD WANTED FOR TREATMENT, SQUARE EDGES:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 5 (*a*+*b*+*c*+*d*+*e*), 9 (*a*+*b*), 15, 19*C*, 20*C*, 22.

No. 15. BEAMS AND STRINGERS, SELECT, SAPWOOD WANTED FOR TREATMENT, WANE PERMITTED:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 5 (*a*+*b*+*c*+*d*+*e*), 9 (*a*+*b*), 15, 19*C*, 20*A*, 22.

Common Beams and Stringers

No. 16. BEAMS AND STRINGERS, COMMON, 85 PER CENT HEARTWOOD, SQUARE EDGES:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 5 (*a*+*b*+*c*+*d*+*e*), 10 (*a*+*b*), 16, 19*D*, 20*C*, 21*B*.

No. 17. BEAMS AND STRINGERS, COMMON, NO HEARTWOOD OR SAPWOOD REQUIREMENT, SQUARE EDGES:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 5 (*a*+*b*+*c*+*d*+*e*), 10 (*a*+*b*), 16, 19*D*, 20*C*.

No. 18. BEAMS AND STRINGERS, COMMON, NO HEARTWOOD OR SAPWOOD REQUIREMENT, WANE PERMITTED:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 5 (*a*+*b*+*c*+*d*+*e*), 10 (*a*+*b*), 16, 19*D*, 20*B*.

No. 19. BEAMS AND STRINGERS, COMMON, SAPWOOD WANTED FOR TREATMENT, SQUARE EDGES:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 5 (*a*+*b*+*c*+*d*+*e*), 10 (*a*+*b*), 16, 19*D*, 20*C*, 22.

No. 20. BEAMS AND STRINGERS, COMMON, SAPWOOD WANTED FOR TREATMENT, WANE PERMITTED:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 5 (*a*+*b*+*c*+*d*+*e*), 10 (*a*+*b*), 16, 19*D*, 20*B*, 22.

Dense Select Joist and Plank

No. 21. JOIST AND PLANK, DOUGLAS FIR OR SOUTHERN PINE, DENSE SELECT, 85 PER CENT HEARTWOOD, SQUARE EDGES:
1*A* (surfaced) or 1*C* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 4 (*a*+*b*+*c*+*d*), 7 (*a*+*b*+*c*), 13, 19*A*, 20*C*, 21*A*, 24, 25, 27, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 22. JOIST AND PLANK, DOUGLAS FIR OR SOUTHERN PINE, DENSE SELECT, NO HEARTWOOD OR SAPWOOD REQUIREMENT, SQUARE EDGES:
1*A* (surfaced) or 1*C* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 4 (*a*+*b*+*c*+*d*), 7 (*a*+*b*+*c*), 13, 19*A*, 20*C*, 24, 25, 27, 28 (*a*+*b*+*c*), Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 23. JOIST AND PLANK, DOUGLAS FIR OR SOUTHERN PINE, DENSE SELECT, NO HEARTWOOD OR SAPWOOD REQUIREMENT, WANE PERMITTED:
1*A* (surfaced) or 1*C* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 4 (*a*+*b*+*c*+*d*), 7 (*a*+*b*+*c*), 13, 19*A*, 20*A*, 24, 25, 27, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 24. JOIST AND PLANK, DOUGLAS FIR OR SOUTHERN PINE, DENSE SELECT, SAPWOOD WANTED FOR TREATMENT, SQUARE EDGES:
1*A* (surfaced) or 1*C* (rough), 2 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 4 (*a*+*b*+*c*+*d*), 7 (*a*+*b*+*c*), 13, 19*A*, 20*C*, 22, 24, 25, 27, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 25. JOIST AND PLANK, DOUGLAS FIR OR SOUTHERN PINE, DENSE SELECT, SAPWOOD WANTED FOR TREATMENT, WANE PERMITTED.
1*A* (surfaced) or 1*C* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 4 (*a*+*b*+*c*+*d*), 7 (*a*+*b*+*c*), 13, 19*A*, 20*A*, 22, 24, 25, 27, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

Select Joist and Plank

No. 26. JOIST AND PLANK, DOUGLAS FIR OR SOUTHERN PINE, SELECT, 85 PER CENT HEARTWOOD, SQUARE EDGES:
1*A* (surfaced) or 1*C* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 4 (*a*+*b*+*c*+*d*), 7 (*a*+*b*+*c*), 13, 19*A*, 20*C*, 21*A*, 23, 25, 26, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 27. JOIST AND PLANK, DOUGLAS FIR OR SOUTHERN PINE, SELECT, NO HEARTWOOD OR SAPWOOD REQUIREMENT, SQUARE EDGES:
1*A* (surfaced) or 1*C* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 4 (*a*+*b*+*c*+*d*), 7 (*a*+*b*+*c*), 13, 19*A*, 20*C*, 23, 25, 26, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 28. JOIST AND PLANK, DOUGLAS FIR OR SOUTHERN PINE, SELECT, NO HEARTWOOD OR SAPWOOD REQUIREMENT, WANE PERMITTED:
1*A* (surfaced) or 1*C* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 4 (*a*+*b*+*c*+*d*), 7 (*a*+*b*+*c*), 13, 19*A*, 20*A*, 23, 25, 26, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 29. JOIST AND PLANK, DOUGLAS FIR OR SOUTHERN PINE, SELECT, SAPWOOD WANTED FOR TREATMENT, SQUARE EDGES:
1*A* (surfaced) or 1*C* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 4 (*a*+*b*+*c*+*d*), 7 (*a*+*b*+*c*), 13, 19*A*, 20*C*, 22, 23, 25, 26, 28, (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 30. JOIST AND PLANK, DOUGLAS FIR OR SOUTHERN PINE, SELECT, SAPWOOD WANTED FOR TREATMENT, WANE PERMITTED:
1*A* (surfaced) or 1*C* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 4 (*a*+*b*+*c*+*d*), 7 (*a*+*b*+*c*), 13, 19*A*, 20*A*, 22, 23, 25, 26, 28 (*a*+*b*+*c*), Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 31. JOIST AND PLANK, SELECT, 85 PER CENT HEARTWOOD, SQUARE EDGES:
1*A* (surfaced) or 1*C* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 4 (*a*+*b*+*c*+*d*), 7 (*a*+*b*+*c*), 13, 19*A*, 20*C*, 21*A*.

No. 32. JOIST AND PLANK, SELECT, NO HEARTWOOD OR SAPWOOD REQUIREMENT, SQUARE EDGES:
1*A* (surfaced) or 1*C* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 4 (*a*+*b*+*c*+*d*), 7 (*a*+*b*+*c*), 13, 19*A*, 20*C*.

No. 33. JOIST AND PLANK, SELECT, NO HEARTWOOD OR SAPWOOD REQUIREMENT, WANE PERMITTED:
1*A* (surfaced) or 1*C* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 4 (*a*+*b*+*c*+*d*), 7 (*a*+*b*+*c*), 13, 19*A*, 20*A*.

No. 34. JOIST AND PLANK, SELECT, SAPWOOD WANTED FOR TREATMENT, SQUARE EDGES:
1*A* (surfaced) or 1*C* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 4 (*a*+*b*+*c*+*d*), 7 (*a*+*b*+*c*), 13, 19*A*, 20*C*, 22.

No. 35. JOIST AND PLANK, SELECT, SAPWOOD WANTED FOR TREATMENT, WANE PERMITTED:
1*A* (surfaced) or 1*C* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 4 (*a*+*b*+*c*+*d*), 7 (*a*+*b*+*c*), 13, 19*A*, 20*A*, 22.

Common Joist and Plank

No. 36. JOIST AND PLANK, COMMON, 85 PER CENT HEARTWOOD, SQUARE EDGES:
1*A* (surfaced) or 1*C* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 4 (*a*+*b*+*c*+*d*), 8 (*a*+*b*+*c*), 14, 19*B*, 20*C*, 21*A*.

No. 37. JOIST AND PLANK, COMMON, NO HEARTWOOD OR SAPWOOD REQUIREMENT, SQUARE EDGES:
1*A* (surfaced) or 1*C* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 4 (*a*+*b*+*c*+*d*), 8 (*a*+*b*+*c*), 14, 19*B*, 20*C*.

No. 38. JOIST AND PLANK, COMMON, NO HEARTWOOD OR SAPWOOD REQUIREMENT, WANE PERMITTED:
1*A* (surfaced) or 1*C* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 4 (*a*+*b*+*c*+*d*), 8 (*a*+*b*+*c*), 14, 19*B*, 20*B*.

No. 39. JOIST AND PLANK, COMMON, SAPWOOD WANTED FOR TREATMENT, SQUARE EDGES:
1*A* (surfaced) or 1*C* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 4 (*a*+*b*+*c*+*d*), *8* (*a*+*b*+*c*), 14, 19*B*, 20*C*, 22.

No. 40. JOIST AND PLANK, COMMON, SAPWOOD WANTED FOR TREATMENT, WANE PERMITTED:
1*A* (surfaced) or 1*C* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 4 (*a*+*b*+*c*+*d*), 8 (*a*+*b*+*c*), 14, 19*B*, 20*B*, 22.

Dense Select Posts and Timbers

No. 41. POSTS AND TIMBERS, DOUGLAS FIR OR SOUTHERN PINE, DENSE SELECT, 85 PER CENT HEARTWOOD, SQUARE EDGES:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 11, (*a*+*b*), 17, 19*E*, 20*C*, 21*C*, 24, 25, 27, 28 (*a*+*b*+*c*), Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 42. POSTS AND TIMBERS, DOUGLAS FIR OR SOUTHERN PINE, DENSE SELECT, NO HEARTWOOD OR SAPWOOD REQUIREMENT, SQUARE EDGES:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 11 (*a*+*b*), 17, 19*E*, 20*C*, 24, 25, 27, 28 (*a*+*b*+*c*), Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 43. POSTS AND TIMBERS, DOUGLAS FIR OR SOUTHERN PINE, DENSE SELECT, NO HEARTWOOD OR SAPWOOD REQUIREMENT, WANE PERMITTED:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 11 (*a*+*b*), 17, 19*E*, 20*A*, 24, 25, 27, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 44. POSTS AND TIMBERS, DOUGLAS FIR OR SOUTHERN PINE, DENSE SELECT, SAPWOOD WANTED FOR TREATMENT, SQUARE EDGES:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 11 (*a*+*b*), 17, 19*E*, 20*C*, 22, 24, 25, 27, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 45. POSTS AND TIMBERS, DOUGLAS FIR OR SOUTHERN PINE, DENSE SELECT, SAPWOOD WANTED FOR TREATMENT, WANE PERMITTED:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 11 (*a*+*b*), 17, 19*E*, 20*A*, 22, 24, 25, 27, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

Select Posts and Timbers

No. 46. POSTS AND TIMBERS, DOUGLAS FIR OR SOUTHERN PINE, SELECT, 85 PER CENT HEARTWOOD, SQUARE EDGES:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 11 (*a*+*b*), 17, 19*E*, 20*C*, 21*C*, 23, 25, 26, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 47. POSTS AND TIMBERS, DOUGLAS FIR OR SOUTHERN PINE, SELECT, NO HEARTWOOD OR SAPWOOD REQUIREMENT, SQUARE EDGES:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 11 (*a*+*b*), 17, 19*E*, 20*C*, 23, 25, 26, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 48. POSTS AND TIMBERS, DOUGLAS FIR OR SOUTHERN PINE, SELECT, NO HEARTWOOD OR SAPWOOD REQUIREMENT, WANE PERMITTED:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 11 (*a*+*b*), 17, 19*E*, 20*A*, 23, 25, 26, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 49. POSTS AND TIMBERS, DOUGLAS FIR OR SOUTHERN PINE, SELECT, SAPWOOD WANTED FOR TREATMENT, SQUARE EDGES:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6 11 (*a*+*b*), 17, 19*E*, 20*C*, 22, 23, 25, 26, 28 (*a*+*b*+*c*), Douglas Fir, or 29 (*a*+*b*+*c*) Southern Pine.

No. 50. POSTS AND TIMBERS, DOUGLAS FIR OR SOUTHERN PINE, SELECT, SAPWOOD WANTED FOR TREATMENT, WANE PERMITTED:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 11 (*a*+*b*), 17, 19*E*, 20*A*, 22, 23, 25, 26, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 51. POSTS AND TIMBERS, SELECT, 85 PER CENT HEARTWOOD, SQUARE EDGES:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 11 (*a*+*b*), 17, 19*E*, 20*C*, 21*C*.

No. 52. POSTS AND TIMBERS, SELECT, NO HEARTWOOD OR SAPWOOD REQUIREMENT, SQUARE EDGES:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 11 (*a*+*b*), 17, 19*E*, 20*C*.

No. 53. POSTS AND TIMBERS, SELECT, NO HEARTWOOD OR SAPWOOD REQUIREMENT, WANE PERMITTED:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 11 (*a*+*b*), 17, 19*E*, 20*A*.

No. 54. POSTS AND TIMBERS, SELECT, SAPWOOD WANTED FOR TREATMENT, SQUARE EDGES:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 11 (*a*+*b*), 17, 19*E*, 20*C*, 22.

No. 55. POSTS AND TIMBERS, SELECT, SAPWOOD WANTED FOR TREATMENT, WANE PERMITTED:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 11 (*a*+*b*), 17, 19*E*, 20*A*, 22.

Common Posts and Timbers

No. 56. POSTS AND TIMBERS, COMMON, 85 PER CENT HEARTWOOD, SQUARE EDGES:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 12 (*a*+*b*), 18, 19*F*, 20*C*, 21*C*.

No. 57. POSTS AND TIMBERS, COMMON, NO HEARTWOOD OR SAPWOOD REQUIREMENT, SQUARE EDGES:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 12 (*a*+*b*), 18, 19*F*, 20*C*.

No. 58. POSTS AND TIMBERS, COMMON, NO HEARTWOOD OR SAPWOOD REQUIREMENT, WANE PERMITTED:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 12 (*a*+*b*), 18, 19*F*, 20*B*.

No. 59. POSTS AND TIMBERS, COMMON, SAPWOOD WANTED FOR TREATMENT, SQUARE EDGES:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 12 (*a*+*b*), 18, 19*F*, 20*C*, 22.

No. 60. POSTS AND TIMBERS, COMMON, SAPWOOD WANTED FOR TREATMENT, WANE PERMITTED.
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 12 (*a*+*b*), 18, 19*F*, 20*B*, 22.

Special Posts and Timbers

No. 61. POSTS AND TIMBERS, DOUGLAS FIR OR SOUTHERN PINE, DENSE SELECT, ONE NARROW FACE ALL HEARTWOOD, OTHER NARROW FACE AND SIDES 85 PER CENT HEARTWOOD, SQUARE EDGES:
1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 11 (*a*+*b*), 17, 19*E*, 20*C*, 21*D*, 24, 25, 27, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 62. POSTS AND TIMBERS, DOUGLAS FIR OR SOUTHERN PINE, SELECT, ONE NARROW FACE ALL HEARTWOOD, OTHER NARROW FACE AND SIDES 85 PER CENT HEARTWOOD, SQUARE EDGES:

1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 11 (*a*+*b*), 17, 19*E*, 20*C*, 21*D*, 23, 25, 26, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 63. POSTS AND TIMBERS, SELECT, ONE NARROW FACE ALL HEARTWOOD, OTHER NARROW FACE AND SIDES 85 PER CENT HEARTWOOD, SQUARE EDGES:

1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 11 (*a*+*b*), 17, 19*E*, 20*C*, 21*D*.

No. 64. POSTS AND TIMBERS, COMMON, ONE NARROW FACE ALL HEARTWOOD, OTHER NARROW FACE AND SIDES 85 PER CENT HEARTWOOD, SQUARE EDGES:

1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 12 (*a*+*b*), 18, 19*F*, 20*C*, 21*D*.

No. 65. POSTS AND TIMBERS, DOUGLAS FIR OR SOUTHERN PINE, DENSE SELECT, ONE NARROW FACE ALL HEARTWOOD, OTHER NARROW AND SIDES 75 PER CENT HEARTWOOD, SQUARE EDGES:

1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 11 (*a*+*b*), 17, 19*E*, 20*C*, 21*E*, 24, 25, 27, 28 (*a*+*b*+*c*), Douglas Fir, or 29 (*a*+*b*+*c*) Southern Pine.

No. 66. POSTS AND TIMBERS, DOUGLAS FIR OR SOUTHERN PINE, SELECT, ONE NARROW FACE ALL HEARTWOOD, OTHER NARROW FACE AND SIDES 75 PER CENT HEARTWOOD, SQUARE EDGES:

1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 11 (*a*+*b*), 17, 19*E*, 20*C*, 21*E*, 23, 25, 26, 28 (*a*+*b*+*c*) Douglas Fir or 29 (*a*+*b*+*c*) Southern Pine.

No. 67. POSTS AND TIMBERS, SELECT, ONE NARROW FACE ALL HEARTWOOD, OTHER NARROW FACE AND SIDES 75 PER CENT HEARTWOOD, SQUARE EDGES:

1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 11 (*a*+*b*), 17, 19*E*, 20*C*, 21*E*.

No. 68. POSTS AND TIMBERS, COMMON, ONE NARROW FACE ALL HEARTWOOD, OTHER NARROW FACE AND SIDES 75 PER CENT HEARTWOOD, SQUARE EDGES:

1*B* (surfaced) or 1*D* (rough), 2, 3 (*a*+*b*+*c*+*d*+*e*+*f*+*g*+*h*+*i*), 6, 12 (*a*+*b*), 18, 19*F*, 20*C*, 21*E*.

APPENDIX[1]

WORKING STRESSES

Notes on Tables of Working Stresses

Authority. 1. The working stresses in the accompanying Table I are recommended by the Forest Products Laboratory, U. S. Forest Service, for structural grades complying with Basic Provisions for Structural Material of American Lumber Standards, including, also, stresses for red and white oak, as the same structural grades can be applied to hardwoods as to soft. In beam and stringer, and post and timber grades, stresses are given only for the species commonly cut to those sizes. Stresses for any other species can be obtained from the Forest Products laboratory.

Minimum Strength Value. 2. Structural grades are developed to insure minimum strength values. The defects permitted in the Common grades provide material having not less than 60 per cent of the strength of green clear wood, and in the Select grades, of 75 per cent, although in Douglas fir and southern pine the stresses recommended in compression and in extreme fiber in bending are 80 per cent of green clear wood strength on account of the limitation on rate of growth.

Exposure. 3. Working values are given for three conditions of exposure during use: (a) Continuously dry, (b) Occasionally wet but quickly dried, (c) More or less continuously damp or wet. Judgment should be exercised as to the values to be used in a particular case.

(*a*) *Continuously Dry.*—Continuously dry contemplates use in interior or protected construction not subject to conditions of excessive dampness or high humidity.

(*b*) *Occasionally Wet.*—Occasionally wet but quickly dried assumes use in such exterior structures as bridges, trestles, grandstands or bleachers, and exposed frame work of open sheds.

(*c*) *Usually Wet.*—More or less continuously damp or wet would apply to material exposed to waves or tidewater, or in contact with earth, or used in a building in portions that would be more or less continuously wet.

Impact. 4. The working values recommended may be used without allowance for impact up to 100 per cent.

Maximum Horizontal Shear. 5. Working values given for horizontal shear are maximum values. The maximum unit horizontal shear at any point in a beam as calculated is one and one-half of the average unit shear obtained by dividing the total shear at that point by the area of the cross-section.

Analysis for Shear Stress. 6. Recognition of all loads in designing for moving loads, or loads concentrated near a support, gives a calculated shearing stress higher than is actually developed. In calculating the shear at one end of a beam, the concentrated loads between that end and a point distant three times the depth of the beam from it may be considered as acting at that point. In moving loads, as on high-

[1] These data are published as the result of cooperative work by several organizations including the Forest Products Laboratory, the American Railway Engineering Association and the A.S.T.M. They do not form a part of the specification as a purchase specification. They are printed with the specification as a convenience to the user so that the data will be immediately available without further reference.

TABLE I.—WORKING STRESSES FOR JOIST AND PLANK AND BEAMS AND STRINGERS[1]

(Pounds per Square Inch)

FOR MATERIAL COMPLYING WITH STRUCTURAL GRADES OF THE AMERICAN SOCIETY FOR TESTING MATERIALS

Continuously Dry

SPECIES	EXTREME FIBER IN BENDING	COMPRESSION PERPENDICULAR TO GRAIN	MAXIMUM HORIZONTAL SHEAR	MODULUS OF ELASTICITY
SELECT GRADE				
CEDAR: western red	900	200	80	1 000 000
northern and southern white	750	175	70	800 000
Port Orford	1100	250	90	1 200 000
Alaska	1100	250	90	1 200 000
CYPRESS: southern	1300	350	100	1 200 000
DOUGLAS FIR:				
Coast Region: Select	1600	345	90	1 600 000
Dense Select	1750	380	105	1 600 000
Rocky Mountain Region	1100	275	85	1 200 000
FIR: balsam	900	150	70	1 000 000
golden, noble, silver, white	1100	300	70	1 100 000
HEMLOCK: West Coast	1300	300	75	1 400 000
eastern	1100	300	70	1 100 000
LARCH: western	1200	325	100	1 300 000
OAK: red and white	1400	500	125	1 500 000
PINE: southern: Select	1600	345	110	1 600 000
Dense Select	1750	380	128	1 600 000
California, Idaho and Northern white, Pondoas and sugar	900	250	85	1 000 000
Norway	1100	300	85	1 200 000
REDWOOD	1200	250	70	1 200 000
SPRUCE: red, white, Sitka	1100	250	85	1 200 000
Englemann	750	175	70	800 000
TAMARACK: eastern	1200	300	95	1 300 000
COMMON GRADE				
CEDAR: western red	720	200	64	1 000 000
northern and southern white	600	175	56	800 000
Port Orford	880	250	72	1 200 000
Alaska	880	250	72	1 200 000
CYPRESS: southern	1040	350	80	1 200 000
DOUGLAS FIR: Coast region	1200	325	72	1 600 000
Rocky Mtn. Region	880	275	68	1 200 000
FIR: balsam	720	150	56	1 000 000
golden, noble, silver, white	880	300	56	1 100 000
HEMLOCK: West Coast	1040	300	60	1 400 000
eastern	880	300	56	1 100 000
LARCH: western	960	325	80	1 300 000
OAK: red and white	1120	500	100	1 500 000
PINE: southern	1200	325	88	1 600 000
California, Idaho and northern white, Pondosa and sugar	720	250	68	1 000 000
Norway	880	300	68	1 200 000
REDWOOD	960	250	56	1 200 000
SPRUCE: red, white, Sitka	880	250	68	1 200 000
Englemann	600	175	56	800 000
TAMARACK: eastern	960	300	76	1 300 000

[1] Values are those recommended by Forest Products Laboratory, U. S. Forest Service.

TABLE I.—WORKING STRESSES[1] (*Continued*)

(Pounds per Square Inch)

Occasionally Wet But Quickly Dried

SPECIES	EXTREME FIBER IN BENDING 4 IN. AND THINNER	EXTREME FIBER IN BENDING 5 IN. AND THICKER	COMPRESSION PERPENDICULAR TO GRAIN	MAXIMUM HORIZONTAL SHEAR	MODULUS OF ELASTICITY
SELECT GRADE					
CEDAR: western red	710	800	150	80	1 000 000
northern and southern white	580	....	140	70	800 000
Port Orford	890	1000	200	90	1 200 000
Alaska	890	....	200	90	1 200 000
CYPRESS: southern	980	....	250	100	1 200 000
DOUGLAS FIR:					
Coast Region: Select	1240	1385	240	90	1 600 000
Dense Select	1370	1515	265	105	1 600 000
Rocky Mtn. Region	800	900	225	85	1 200 000
FIR: balsam	670	....	125	70	1 000 000
golden, noble, silver, white	800	....	225	70	1 100 000
HEMLOCK: West Coast	980	1100	225	75	1 400 000
eastern	800	....	225	70	1 100 000
LARCH: western	980	1100	225	100	1 300 000
OAK: red and white	1070	1200	375	125	1 500 000
PINE: southern: Select	1240	1385	240	110	1 600 000
Dense Select	1370	1515	265	128	1 600 000
California, Idaho and northern white, Pondosa and sugar	710	....	150	85	1 000 000
Norway	890	....	175	85	1 200 000
REDWOOD	890	1000	150	70	1 200 000
SPRUCE: red, white, Sitka	800	900	150	85	1 200 000
Englemann	580	....	140	70	800 000
TAMARACK: eastern	980	....	225	95	1 300 000
COMMON GRADE					
CEDAR: western red	600	640	150	64	1 000 000
northern and southern white	490	....	140	56	800 000
Port Orford	760	800	200	72	1 200 000
Alaska	760	....	200	72	1 200 000
CYPRESS: southern	830	....	250	80	1 200 000
DOUGLAS FIR: Coast region	980	1040	225	72	1 600 000
Rocky Mountain region	680	720	225	68	1 200 000
FIR: balsam	570	....	125	56	1 000 000
golden, noble, silver, white	680	....	225	56	1 100 000
HEMLOCK: West Coast	830	880	225	60	1 400 000
eastern	680	....	225	56	1 100 000
LARCH: western	830	880	225	80	1 300 000
OAK: red and white	900	960	375	100	1 500 000
PINE: southern	980	1040	225	88	1 600 000
California, Idaho and northern white, Pondosa and sugar	600	....	150	68	1 000 000
Norway	760	....	175	68	1 200 000
REDWOOD	760	800	150	56	1 200 000
SPRUCE: red, white, Sitka	680	720	150	68	1 200 000
Englemann	490	....	140	56	800 000
TAMARACK: eastern	830	....	225	76	1 300 000

[1] Values are those recommended by Forest Products Laboratory, U. S. Forest Service.

TABLE I.—WORKING STRESSES[1] (*Continued*)

(Pounds per Square Inch)

More or Less Continuously Damp or Wet

SPECIES	EXTREME FIBER IN BENDING 4 IN. AND THINNER	EXTREME FIBER IN BENDING 5 IN. AND THICKER	COMPRESSION PERPENDICULAR TO GRAIN	MAXIMUM HORIZONTAL SHEAR	MODULUS OF ELASTICITY
SELECT GRADE					
CEDAR: western red	670	750	125	80	1 000 000
northern and southern white	530	....	100	70	800 000
Port Orford	800	900	150	90	1 200 000
Alaska	800	....	150	90	1 200 000
CYPRESS: southern	800	....	225	100	1 200 000
DOUGLAS FIR:					
Coast Region: Select	950	1065	215	90	1 600 000
Dense Select	1050	1165	235	105	1 600 000
Rocky Mtn. Region	620	700	200	85	1 200 000
FIR: balsam	530	....	100	70	1 000 000
golden, noble, silver, white	710	....	200	70	1 100 000
HEMLOCK: West Coast	800	900	200	75	1 400 000
eastern	710	....	200	70	1 100 000
LARCH: western	800	900	200	100	1 300 000
OAK: red and white	890	1000	300	125	1 500 000
PINE: southern: Select	950	1065	215	110	1 600 000
Dense Select	1050	1165	235	128	1 600 000
California, Idaho and northern white, Pondosa and sugar	670	....	125	85	1 000 000
Norway	710	....	150	85	1 200 000
REDWOOD	710	800	125	70	1 200 000
SPRUCE: red, white, Sitka	710	800	125	85	1 200 000
Englemann	440	....	100	70	800 000
TAMARACK: eastern	800	....	200	95	1 300 000
COMMON GRADE					
CEDAR: western red	570	600	125	64	1 000 000
northern and southern white	450	....	100	56	800 000
Port Orford	680	720	150	72	1 200 000
Alaska	680	....	150	72	1 200 000
CYPRESS: southern	680	....	225	80	1 200 000
DOUGLAS FIR: Coast region	750	800	200	72	1 600 000
Rocky Mountain region	530	560	200	68	1 200 000
FIR: balsam	450	....	100	56	1 000 000
golden, noble, silver, white	600	....	200	56	1 100 000
HEMLOCK: West Coast	680	720	200	60	1 400 000
eastern	600	....	200	56	1 100 000
LARCH: western	680	720	200	80	1 300 000
OAK: red and white	750	800	300	100	1 500 000
PINE: southern	750	800	200	88	1 600 000
California, Idaho and northern white, Pondosa and sugar	570	....	125	68	1 000 000
Norway	600	....	150	68	1 200 000
REDWOOD	600	640	125	56	1 200 000
SPRUCE: red, white, Sitka	600	640	125	68	1 200 000
Englemann	370	....	100	56	800 000
TAMARACK: eastern	680	....	200	76	1 300 000

[1] Values are those recommended by Forest Products Laboratory, U. S. Forest Service.

way bridges or railway stringers, in computing the shear at one end it is safe to ignore all wheel loads between that end and a point three times the depth of the beam or stringer from it, when the balance of the span is assumed loaded so as to give a maximum shear stress.

Shear Stresses for Joint Details.

7. Shear stresses for joint details may be taken as 50 per cent greater than the values for horizontal shear given in Table I.

Permanent Set.

8. Timber constantly yields under long continued loading, acquiring a permanent set. This set with a fully loaded beam is about equal to the deflection using the modulus of elasticity as given in the tables. In order to minimize the results of sag, it is advisable to use values one-half those given in the tables.

Compression in Short Columns.

9. The working stresses for compression parallel to grain are for use on posts, struts, etc., with unsupported length not greater than ten times their least dimension. They are also for use in end bearing on compression members, as a short column or strut is more likely to fail at the end than at any other point in its length, and the variations in moisture content are greater there.

Compression in Medium Length Columns.

10. For columns of intermediate length, the Forest Products Laboratory finds from tests recently made that a fourth-power parabola, tangent to the Euler curve, is a conservative representation of the law controlling the strength. That is, from the short block to the long column in which the strength is dependent on stiffness, there is a falling off in ultimate strength which follows a smooth curve, very flat at first but curving sharply to become tangent to the Euler curve at two-thirds of the ultimate crushing strength.

Formula.

11. For columns from $\frac{P}{A} = S$ to $\frac{P}{A} = \frac{2S}{3}$,

$$\frac{P}{A} = S\left[1 - \frac{1}{3}\left(\frac{l}{Kd}\right)^4\right]$$

where P = total load in pounds;
A = area in square inches;
$\frac{P}{A}$ = unit compressive stress;
S = Safe stress in compression parallel to grain for short columns;
l = unsupported length in inches;
d = least dimension in inches;
E = modulus of elasticity;
K = The $\frac{l}{d}$ at the point of tangency of the parabolic and Euler curves, at which $\frac{P}{A} = \frac{2S}{3}$. The value of K for any species and grade is: $\frac{\pi}{2}\sqrt{\frac{E}{6S}}$.

Influence of Defects.

12. The influence of defects on the compressive strength of columns of constant-cross section decreases as the length increases. When $\frac{l}{d}$ equals the value of K for the species and grade, defects such as are allowable in the grade have little influence on the strength as a column. Beyond this length the investigation of the strength of columns indicated that the Euler formula is quite accurate for long wooden columns with pin-end connections and that the

maximum load is dependent upon stiffness. In such columns, a factor of safety of 3 should be applied to values of modulus of elasticity in order to obtain safe loading.

Influence of End Conditions.

13. The laboratory does not, with the present data and under ordinary conditions, find justification for increasing the stresses on square-end columns over those for carefully-centered pin-end columns. Tests to determine the influence of end conditions are still being made and it is probable that under special conditions higher stresses can be used.

Long Columns.

14. For long columns, with factor of safety of 3, $\frac{P}{A} = \frac{\pi^2}{36} \frac{E}{\left(\frac{l}{d}\right)^2}$.

Maximum Length.

15. Columns should be limited in slenderness to $\frac{l}{d} = 50$.

Direct Tension.

16. For direct tension the same values as for extreme fiber stress in bending may be used. Straight-grained wood has greater resistance to tension than to any other kind of stress. It has been found, however, practically impossible to design joints that will develop anywhere near the full tensile strength.

Joists and Beams in Direct Tension.

17. Grades of joists or beams may be used for members in direct tension, as in bottom chords of trusses, increase in size of defects towards ends being permissible because of the gradual application of stresses through splice plates or end connections.

Joist and Plank, Vertical or Horizontal.

18. The provisions of the joist and plank grades are such that working stresses for these grades may be applied to material used with wide faces vertical or horizontal. In material 5 in. and thicker with loads applied to the wide face, the knot requirements for this face are those for the narrow face as given in the rules.

Working Stresses in Timbers Nearly Square.

19. Where working stresses in bending are required for caps, bridge ties, etc., they should be graded on beam and stringer grades, but as such material is often square or has horizontal faces wider than the vertical faces, in contrast to beams and stringers in which the narrow faces are horizontal and the wide faces are vertical, care should be exercised that the knot limitations are applied to the proper faces.

Two-Span Stringers.

20. In railway stringers of two spans length, defects throughout the center two-thirds should be limited as in the center third of single span stringers, for the maximum moment will be over the center support and although the full positive moment would not be developed in either span as long as there was resistance to negative moment over the center support, there might be circumstances in which full positive moment of resistance at the centers of the two spans would be desirable.

FACTORS OF SAFETY

Elastic Limit and Breaking Strength.

21. In determining working stresses, the Forest Products Laboratory has considered both elastic limit and breaking strength. Elastic limit, however, is more variable and less definite than ultimate strength, and the latter is taken as the more dependable basis for the determination of safe working stresses.

Factor of Safety.

22. The factor of safety at a given working stress varies materially with the duration of the stress. At the recommended working stresses, given in the table for continuously dry locations, the average timber in buildings has a

factor of safety of 6 on impact loading,[1] a factor of 4 for five-minute loads and 2½ for long-time loading, with a minimum factor of safety of 2 on 75 per cent of the pieces under long-time loading. About one piece in 100, of very light weight and with maximum defects for the grade, would be expected to break at 1½ times the recommended stress under loading of approximately 10 years duration. For new timbers in bridge construction, the factors of safety are about 15 per cent greater.

FACTORS AFFECTING WORKING STRESSES

Basis of Working Strength.

23. Working stresses are based on the strength of the clear wood of the various species and, in some properties, on grade as fixed by limitation on size and location of knots, extent of shake and checks and extent of cross grain, on conditions of exposure during use, and on size of piece. In southern pine and Douglas fir working stresses in some properties are increased for limitation on rate of growth and for requirement of percentage of summerwood.

Variations With Exposure, Grade, Size.

24. Working stresses for extreme fiber in bending are varied with grade, extent of exposure, and size of piece; in horizontal shear, with grade; in compression parallel to grain, with grade and exposure; in compression perpendicular to grain, with exposure.

25. Working stresses in shear are not varied with size or extent of exposure; in compression parallel to grain they are not varied with size; in compression perpendicular to grain they are not varied with grade or size; in modulus of elasticity they are taken as the same in all grades.

Rate of Growth and Density.

26. In southern pine and Douglas fir, working stresses in extreme fiber in bending, compression parallel to grain, and compression perpendicular to grain, in any grade, may be increased one-fifteenth for material of close grain, or one-sixth for dense material, over material not so selected. Values in these species in shear may be increased one-sixth for dense material, but not for limitation on rate of growth. Modulus of elasticity is not varied with these properties.

Variations in Working Stresses.

27. The several permitted working stresses vary with conditions as follows, a √ indicating that the stress varies and a blank (..) indicating the stress does not vary:

PROPERTY	GOVERNING DEFECTS	CONDITIONS OF EXPOSURE	GRADE	SIZE OF PIECE	RATE OF GROWTH	DENSITY
Extreme fiber stress in bending	Knots and Angle of Grain	√	√	√[a]	√	√
Horizontal shear	Shakes and Checks	..	√	..	..	√
Compression parallel to Grain	Knots and Angle of Grain	√	√	..	√	√
Compression Perpendicular to Grain		√	..	..	√	√
Modulus of Elasticity		..	..	..	..	..

[a] Dry location only.

[1] If impact stresses are neglected when less than 100 per cent of the live load producing them, the factor of safety for such loads would be reduced from 6 to a minimum of 3.

TABLE II.—WORKING STRESSES FOR POSTS AND TIMBERS 6 BY 6 IN. AND LARGER[1]

(Pounds per Square Inch)

FOR MATERIAL COMPLYING WITH STRUCTURAL GRADES OF THE AMERICAN SOCIETY FOR TESTING MATERIALS

Continuously Dry

SPECIES	COMPRESSION PARALLEL TO GRAIN RATIO OF LENGTH TO LEAST DIMENSION (L/D)										MODULUS OF ELASTICITY
	10	12	14	16	18	20	25	30	35	40	50	
SELECT GRADE												
CEDAR: western red	700	686	674	656	629	592	438	304	224	171	110	1 000 000
DOUGLAS FIR:												
Coast region: Select	1175	1149	1127	1093	1045	975	702	487	358	274	175	1 600 000
Dense Select	1285	1251	1222	1176	1112	1022	702	487	358	274	175	1 600 000
Rocky Mtn. region	800	786	774	753	726	688	526	365	268	206	132	1 200 000
HEMLOCK: West Coast	900	885	872	852	823	783	614	426	313	240	153	1 400 000
LARCH: western	1100	1068	1041	999	937	851	570	396	291	223	142	1 300 000
OAK: red and white	1000	982	967	943	908	860	658	457	336	257	164	1 500 000
PINE: southern: Select	1175	1149	1127	1093	1045	975	702	487	358	274	175	1 600 000
Dense Select	1285	1251	1222	1176	1112	1022	702	487	358	274	175	1 600 000
REDWOOD	1000	972	947	910	856	781	526	365	268	206	132	1 200 000
SPRUCE: red, white, Sitka	800	786	774	753	726	688	526	365	268	206	132	1 200 000
COMMON GRADE												
CEDAR: western red	560	553	547	538	524	505	425	304	224	171	110	1 000 000
DOUGLAS FIR: Coast region	880	870	861	847	826	796	675	487	358	274	175	1 600 000
Rocky Mtn. Region	640	632	627	617	602	582	500	365	268	206	132	1 200 000
HEMLOCK: West Coast	720	712	706	696	680	660	573	426	313	240	153	1 400 000
LARCH: western	880	863	849	828	798	752	570	396	291	223	142	1 300 000
OAK: red and white	800	790	783	771	753	728	625	457	336	257	164	1 500 000
PINE: southern	880	870	861	847	826	796	675	487	358	274	175	1 600 000
REDWOOD	800	786	773	754	726	688	526	365	268	206	132	1 200 000
SPRUCE: red, white, Sitka	640	632	627	617	602	582	500	365	268	206	132	1 200 000

[1] Values are those recommended by Forest Products Laboratory, U. S. Forest Service.

TABLE II.—WORKING STRESSES[1] (*Continued*)

(Pounds per Square Inch)

FOR MATERIAL COMPLYING WITH STRUCTURAL GRADES OF THE AMERICAN SOCIETY FOR TESTING MATERIALS

POSTS AND TIMBERS 6 BY 6 IN. AND LARGER

Occasionally Wet But Quickly Dried

SPECIES	COMPRESSION PARALLEL TO GRAIN RATIO OF LENGTH TO LEAST DIMENSION (l/d)											MODULUS OF ELASTICITY
	10	12	14	16	18	20	25	30	35	40	50	
SELECT GRADE												
CEDAR: western red	700	686	673	654	628	591	438	304	224	171	110	1 000 000
DOUGLAS FIR:												
Coast region: Select	1065	1045	1028	1003	968	915	702	487	358	274	175	1 600 000
Dense Select	1165	1139	1118	1083	1036	971	702	487	358	274	175	1 600 000
Rocky Mtn. region	800	785	772	753	728	688	526	365	268	206	132	1 200 000
HEMLOCK: West Coast	900	885	871	851	824	783	612	426	313	240	153	1 400 000
LARCH: western	1000	976	955	922	877	810	570	396	291	223	142	1 300 000
OAK: red and white	900	886	876	859	834	798	652	457	336	257	164	1 500 000
PINE: southern: Select	1065	1045	1028	1003	968	915	702	487	358	274	175	1 600 000
Dense Select	1165	1139	1118	1083	1036	971	702	487	358	274	175	1 600 000
REDWOOD	900	879	861	834	794	738	526	365	268	206	132	1 200 000
SPRUCE: red, white, Sitka	750	738	728	712	690	657	525	365	268	206	132	1 200 000
COMMON GRADE												
CEDAR: western red	560	552	546	537	523	504	425	304	224	171	110	1 000 000
DOUGLAS FIR: Coast region	800	792	784	773	758	736	644	487	358	274	175	1 600 000
Rocky Mtn. region	640	632	625	616	602	582	502	365	268	206	132	1 200 000
HEMLOCK: West Coast	720	712	705	695	681	659	572	426	313	240	153	1 400 000
LARCH: western	800	787	777	760	736	704	564	396	291	223	142	1 300 000
OAK: red and white	720	714	708	698	686	668	592	457	336	257	164	1 500 000
PINE: southern	800	792	784	773	758	736	644	487	358	274	175	1 600 000
REDWOOD	720	709	700	685	666	637	518	365	268	206	132	1 200 000
SPRUCE: red, white, Sitka	600	594	588	580	568	552	485	365	268	206	132	1 200 000

[1] Values are those recommended by Forest Products Laboratory, U. S. Forest Service.

TABLE II.—WORKING STRESSES[1] (*Continued*)

(Pounds per Square Inch)

FOR MATERIAL COMPLYING WITH STRUCTURAL GRADES OF THE AMERICAN SOCIETY FOR TESTING MATERIALS

POSTS AND TIMBERS 6 BY 6 IN. AND LARGER

More or Less Continuously Damp or Wet

SPECIES	COMPRESSION PARALLEL TO GRAIN RATIO OF LENGTH TO LEAST DIMENSION (l/d)											MODULUS OF ELASTICITY
	10	12	14	16	18	20	25	30	35	40	50	
SELECT GRADE												
CEDAR: western, red	650	638	629	614	594	565	437	304	224	171	110	1 000 000
DOUGLAS FIR:												
Coast region: Select	905	893	883	867	846	814	683	487	358	274	175	1 600 000
Dense Select	990	974	961	940	910	871	698	487	358	274	175	1 600 000
Rocky Mtn. region	700	690	681	669	651	623	514	365	268	206	132	1 200 000
HEMLOCK: West Coast	800	789	780	766	745	717	600	426	313	240	153	1 400 000
LARCH: western	800	787	776	760	736	704	565	396	291	223	142	1 300 000
OAK: red and white	800	790	783	771	753	728	625	457	336	257	164	1 500 000
PINE: southern: Select	905	893	883	867	846	814	683	487	358	274	175	1 600 000
Dense Select	990	974	961	940	910	871	698	487	358	274	175	1 600 000
REDWOOD	750	737	727	712	690	657	525	365	268	206	132	1 200 000
SPRUCE: red, white, Sitka	650	642	635	625	611	589	500	365	268	206	132	1 200 000
COMMON GRADE												
CEDAR: western red	520	514	509	502	491	475	413	304	224	171	110	1 000 000
DOUGLAS FIR: Coast region	680	675	670	664	655	641	588	482	358	274	175	1 600 000
Rocky Mtn. region	560	554	551	544	535	521	465	365	268	206	132	1 200 000
HEMLOCK: West Coast	640	634	629	622	612	598	537	426	313	240	153	1 400 000
LARCH: western	640	633	627	618	606	588	519	396	291	223	142	1 300 000
OAK: red and white	640	636	631	625	616	603	550	453	336	257	164	1 500 000
PINE: southern	680	675	670	664	655	641	588	482	358	274	175	1 600 000
REDWOOD	600	594	588	580	568	552	483	365	268	206	132	1 200 000
SPRUCE: red, white, Sitka	520	515	512	507	500	489	446	365	268	206	132	1 200 000

[1] Values are those recommended by Forest Products Laboratory, U. S. Forest Service.

SAFE LOADS FOR WOODEN COLUMNS

1. The unit working stresses in compression parallel to grain for columns whose ratio of unsupported length to least dimension does not exceed 10 shall be not greater than that given for the species in the accompanying Table II on working stresses.

2. For columns the ratio of whose unsupported length to least dimension is greater than 10, the following formula shall be used until the reduction in allowable stress equals one-third the stress for short columns:

$$\frac{P}{A} = S\left[1 - \frac{1}{3}\left(\frac{l}{Kd}\right)^4\right]$$

where P = total load in pounds;
A = area in square inches;
$\frac{P}{A}$ = unit compressive stress;
S = safe stress in compression parallel to grain for short columns;
l = unsupported length in inches;
d = least dimension in inches;
E = modulus of elasticity;
K = the $\frac{l}{d}$ at the point of tangency of the parabolic and Euler curves, at which $\frac{P}{A} = \frac{2}{3}S$.

The value of K for any species and grade is $\frac{\pi}{2}\sqrt{\frac{E}{6S}}$.

3. For columns of greater length, the Euler formula below, which includes a factor of safety of 3, shall be used:

$$\frac{P}{A} = \frac{\pi^2}{36}\frac{E}{\left(\frac{l}{d}\right)^2}$$

4. Columns shall be limited in slenderness to $\frac{l}{d} = 50$.

VALUES OF K FOR COLUMNS OF INTERMEDIATE LENGTH

Species	Continuously Dry		Occasionally Wet		Usually Wet	
	Select	Common	Select	Common	Select	Common
Cedar, western red	24.2	27.1	24.2	27.1	25.1	28.1
Cedar, Port Orford	23.4	26.2	24.6	27.4	25.6	28.7
Douglas Fir, Coast region: Select	23.7	27.3	24.9	28.6	27.0	31.1
Douglas Fir, Dense Select	22.6	24.9	23.8	26.1	25.8	28.3
Douglas Fir, Rocky Mountain region	24.8	27.8	24.8	27.8	26.5	29.7

VALUES OF K FOR COLUMNS OF INTERMEDIATE LENGTH—*Continued*

SPECIES	CONTINUOUSLY DRY		OCCASIONALLY WET		USUALLY WET	
	SELECT	COMMON	SELECT	COMMON	SELECT	COMMON
Hemlock, West Coast	25.3	28.3	25.3	28.3	26.8	30.0
Larch, western	22.0	24.6	23.1	25.8	25.8	28.8
Oak, red and white	24.8	27.8	26.1	29.3	27.7	31.1
Pine, southern: Select	23.7	27.3	24.9	28.6	27.0	31.1
Pine, Dense Select	22.6	24.9	23.8	26.1	25.8	28.3
Redwood	22.2	24.8	23.4	26.1	25.6	28.6
Spruce, red, white, Sitka	24.8	27.8	25.6	28.7	27.5	30.8

STRENGTH OF COLUMNS OF INTERMEDIATE LENGTH, EXPRESSED AS A PERCENTAGE OF STRENGTH OF SHORT COLUMNS

Values for the expression $\left[1-\frac{1}{3}\left(\frac{l}{Kd}\right)^4\right]$ in the formula: $\frac{P}{A}=S\left[1-\frac{1}{3}\left(\frac{l}{Kd}\right)^4\right]$

	RATIO OF LENGTH TO LEAST DIMENSION IN RECTANGULAR TIMBERS $\frac{l}{d}$																			
K	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31
22	97	96	95	93	91	88	85	81	77	72	67	..	..	..	..	..	..	..	..	..
23	98	97	95	94	92	90	87	84	81	77	72	67	..	..	..	..	..	..	..	..
24	98	97	96	95	93	92	89	87	84	80	76	72	67	..	..	..	..	..	..	..
25	98	98	97	96	94	93	91	89	86	83	80	76	72	67	..	..	..	..	..	..
26	99	98	97	96	95	93	92	91	89	86	83	80	76	72	67	..	..	..	..	..
27	99	98	98	97	96	95	93	92	90	88	85	82	79	74	71	67	..	..	..	..
28	99	98	98	97	96	95	94	93	91	89	87	85	82	79	75	71	67	..	..	..
29	99	99	98	98	97	96	95	94	92	91	89	87	84	82	79	75	71	67	..	..
30	99	99	98	98	97	97	96	95	94	92	90	88	86	84	81	78	75	71	67	..
31	99	99	99	98	98	97	96	95	94	93	92	90	88	86	84	81	78	75	71	67

NOTE.—This table can also be used for columns not rectangular, the l/d being equivalent to 0.289 l/r, where r is the least radius of gyration of the section.

STANDARD SPECIFICATIONS
FOR
SOUTHERN YELLOW-PINE PILES AND POLES TO BE CREOSOTED[1]

Serial Designation: D 25 – 20

These specifications are issued under the fixed designation D 25; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1915; ADOPTED, 1920.

General Requirements.

1. The specifications as to strength shall agree with the requirements of the Standard Definitions of Terms Relating to Structural Timber (Serial Designation: D 9) of the American Society for Testing Materials.[2]

Sapwood.

2. All piles or telegraph poles shall show 40 per cent sapwood in cross-section, or there shall be a ring of sapwood not less than 1 in. in thickness all around the heartwood.

Quality.

3. (*a*) Piles and poles shall be cut from sound live trees, of straight grain and regular taper; without crooks exceeding one-fourth the diameter of the stick at middle of crook when peeled. They shall be free from rot, red heart, holes or rotten knots, shakes and felling checks.

(*b*) All piles and poles shall have the bark and inner skin carefully removed when the tree is felled; all limbs and knots trimmed flush and butts cut square.

Minimum Diameter.

4. The minimum diameter of piles after peeling shall be as follows:

LENGTH.	BUTTS, IN.	TOPS, IN.
36 ft. and under	14	10
38 ft. and under 50 ft	14	9
50 ft. and over	15	9

No pile with butt diameter over 18 in., nor top diameter over 13½ in., will be accepted. The length of each pile shall be legibly marked on the butt with white or black paint.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

[2] See p. 697.

STANDARD SPECIFICATIONS FOR WOODEN PAVING BLOCKS FOR EXPOSED PAVEMENTS[1]

Serial Designation: D 52 – 20

These specifications are issued under the fixed designation D 52; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1918; ADOPTED IN AMENDED FORM, 1920.

Scope.

1. These specifications cover wooden paving blocks for pavements exposed to alternate wet and dry conditions, as distinguished from pavements which are used under cover and protected from atmospheric influences.

TIMBER

Kind of Timber.

2. The wood, which shall be treated, shall be Southern yellow pine, Douglas fir, tamarack, Norway pine, hemlock, or black gum. Only one kind of wood shall be used in any one contract. The blocks shall be sound, and shall be well manufactured, square-butted, square-edged, free from unsound, loose or hollow knots, knot holes, worm holes, and other defects such as shakes, checks, etc., that would be detrimental to the blocks.

Quality of Timber.

3. The number of annual rings in the one inch which begins two inches from the pith of the block shall not be less than six, measured radially; provided, however, that blocks containing between five and six rings in this inch shall be accepted if they contain 33⅓ per cent or more summerwood. In case the block does not contain the pith, the one inch to be used shall begin one inch away from the ring which is nearest to the heart of the block. The blocks in each charge shall contain an average of at least 70 per cent of heartwood. No one block shall be accepted that contains less than 50 per cent of heartwood.

Size of Blocks.

4. The blocks shall be from 5 to 10 in. long, but should preferably average two times the depth; they shall be .....in. in depth.[2] They may be from 3 to 4 in. in width, but in any

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

[2] It is recommended that blocks 4 in. in depth be used for streets with very heavy traffic, and blocks 3½ in. in depth for streets with moderate traffic. For streets with light traffic, blocks 3 in. in depth may be used, but where 3-in. blocks are used no block shall be longer than 8 in.

one city block all of them shall be of uniform width. A variation of $\frac{1}{16}$ in. shall be allowed in the depth and $\frac{1}{8}$ in. in the width of the blocks from that specified. In all cases the width shall be greater or less than the depth by at least $\frac{1}{4}$ in.

TREATMENT

Treatment.

5. The timber may be either air-seasoned or green, but should preferably be treated within three months from the time it is sawed. Green timber and seasoned timber shall not, however, be treated together in the same charge. The blocks shall be treated in an air-tight cylinder with the preservative specified by the purchaser. In all cases, whether thoroughly air-seasoned or green, they shall first be subjected to live steam at a temperature between 220 and 240° F.[1] for not less than 2 hours nor more than 4 hours, at the discretion of the treating plant operator, after which they shall be subjected to a vacuum of not less than 22 in. held for at least one hour. While the vacuum is still on, the preservative oil, heated to a temperature of between 180 and 220° F., shall be run in until the cylinder is completely filled, care being taken that no air is admitted. Pressure shall then be gradually applied not to exceed 50 lb. at the end of the first hour nor 100 lb. at the end of the second hour, and then maintained at not less than 100 lb. nor more than 150 lb. until the wood has absorbed the required amount of oil.[2]

After this a supplemental vacuum, in which the maximum intensity reached is at least 20 in. and the time the vacuum is applied not less than 30 minutes, shall be applied. If desired, this vacuum may be followed by a short steaming period.

Penetration of Preservative.

6. In any charge blocks shall contain at least 16 lb. of water-free oil per cubic foot of wood at the completion of the treatment. The blocks after treatment shall show satisfactory penetration of the preservative, and in all cases the oil shall be diffused throughout the sap wood. To determine this, at least 25 blocks shall be selected from various parts of each charge and sawed in half perpendicular to the fibers through the center, and if more than one of these blocks show untreated sapwood, the charge shall be re-treated. After re-treating, the charge shall be again subjected to a similar inspection.

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[1] In no case shall a steam pressure of 20 lb. per sq. in. be exceeded.

[2] This treatment is recommended for yellow pine only. It is probably also suited to Norway pine, hemlock, black gum and tamarack, but not to Douglas fir. Further recommendations on the treatment of these species are reserved for the future.

Handling Blocks After Treatment.

7. Blocks shall preferably be laid in the street as soon as possible after being treated. If they cannot be laid immediately, provision shall be made to prevent them from drying out by stacking in close piles and covering them, and, if possible, by sprinkling them thoroughly at intervals. In any case, where they are not laid as soon as they are received on the street, they shall be well sprinkled about two days before being laid, under the direction of the purchaser. It is important to have the wood sufficiently wet to be swelled to its maximum size before it is laid.

PRESERVATIVE

Preservative.

8. The preservative used shall be coal-tar paving oil or distillate oil, as specified by the purchaser.

INSPECTION

Inspection at Plant.

9. All material herein specified and processes used in the manufacture of the blocks therefrom shall be subject to inspection, acceptance, or rejection at the plant of the manufacturer, which shall be equipped with all the necessary gages, appliances, and facilities to enable the inspector to satisfy himself that the requirements of the specifications are being fulfilled.

Inspection at Street.

10. The purchaser shall have the further right to inspect the blocks after delivery upon the street, for the purpose of rejecting any blocks that do not meet these specifications; except that the plant inspections shall be final with respect to the kind of wood, number of rings per inch, oil, and treatment.

STANDARD METHODS OF TESTING SMALL CLEAR SPECIMENS OF TIMBER[1]

Serial Designation: D 143 – 27

These methods are issued under the fixed designation D 143; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED IN AMENDED FORM, 1927.

The everyday use of timber for multitudinous purposes makes manifest a continual need of data on its mechanical properties. The great variety of species, the variability of the material, the continually changing conditions of supply, the many factors affecting test results, all combine to make the technique of testing wood unique in its complexity.

In the preparation of this procedure for testing small clear specimens, consideration was given both to the desirability of adopting methods which would yield results comparable to those already available and to the possibility of embodying such improvements as experience has shown desirable. In view of the many thousands of tests made under a single comprehensive plan by the U. S. Forest Service, the Forestry Branch of the Department of the Interior, Canada, and other similar organizations, the procedure naturally conforms closely to the methods used by these institutions. This procedure is the outgrowth of a study of both American and European experience and methods. Its general adoption will tend toward a world-wide unification of results, permitting an interchange and correlation of data, and will establish the basis for a cumulative body of fundamental information on the timber species of the world.

The methods under consideration represent the entire procedure from selection of the trees to the manipulation of the test, thus controlling factors, such as the size and proportion of test specimens and rate of loading, which may influence results. No attempt has been made to cover methods of computation and analysis, as these questions may be considered independently at any time. Such sample data and computation sheets and cards have been incorporated, however, as were thought to be of assistance to the investigator in systematizing records.

Purpose of Tests.

1. Tests on small clear specimens of wood are made to afford:

(*a*) Data for comparing the mechanical properties of various species.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

(*b*) Data for the establishment of correct strength functions which, in conjunction with results of tests of timbers in structural sizes,[1] afford the basis for fixing allowable stresses.

(*c*) Data upon which to determine the influence on the mechanical properties of such factors as density, locality of growth, position in cross-section, height of timber in the tree, change of properties with seasoning, and change from sapwood to heartwood.

Kind of Tests.

2. The principal mechanical tests are static bending, compression parallel to grain, impact bending, compression perpendicular to grain, hardness, shear parallel to grain,[2] tension perpendicular to grain, cleavage, and tension parallel to grain. These tests are made on both green and air-dry material as hereinafter specified. In addition, such physical properties as specific gravity, radial shrinkage, tangential shrinkage, and shrinkage in volume are determined.

MATERIAL

COLLECTION

(*A*) *Selection.*

Authentic Identification.

3. The material shall be from trees selected in the forest by one qualified to indentify the species.

Selection and Number of Trees.

4. For each species to be tested from a given locality, at least five representative trees of merchantable size and of approximately average age shall be selected.

Selection and Number of Bolts.

5. The material from five trees of each species from a given locality shall be selected as follows:

(*a*) From one tree, the entire merchantable bole, if not over 16 ft. in length;[3] if over 16 ft. in length, four 4-ft. sections or bolts, the first to be taken at the stump, the second and third at distances of one-eighth and three-eighths, respectively, of the merchantable length above the stump or as near thereto as possible; and the fourth at the top of the merchantable length.

(*b*) From two trees, the 8-ft. section next above the 8-ft. butt log.

(*c*) From two trees, the 4-ft. section next above the 12-ft. butt log.

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[1] See Standard Methods of Conducting Static Tests of Timbers in Structural Sizes (Serial Designation: D 198) of the American Society for Testing Materials, p. 664.

[2] The test for shearing strength perpendicular to the grain (sometimes termed "vertical shear") is not included as one of the principal mechanical tests since in such a test the strength is limited by the shearing resistance parallel to the grain.

[3] This tree is intended to furnish test material for studying, among other things, the variation of properties with height in trees and prrovides for this purpose a 4-ft. section from different heights Experience has shown that four sections or bolts, each 4 ft. in length, will usually be sufficient for studying the influence of height in tree on the mechanical properties.

6. In cases where the logs or bolts are over 36 in. in diameter, a single flitch 6 in. in thickness, taken through the pith in a north and south direction and representing the full diameter of the log, may be substituted, in the same length, for the full log or bolt specified in Section 5. **Substitution of Flitches for Bolts.**

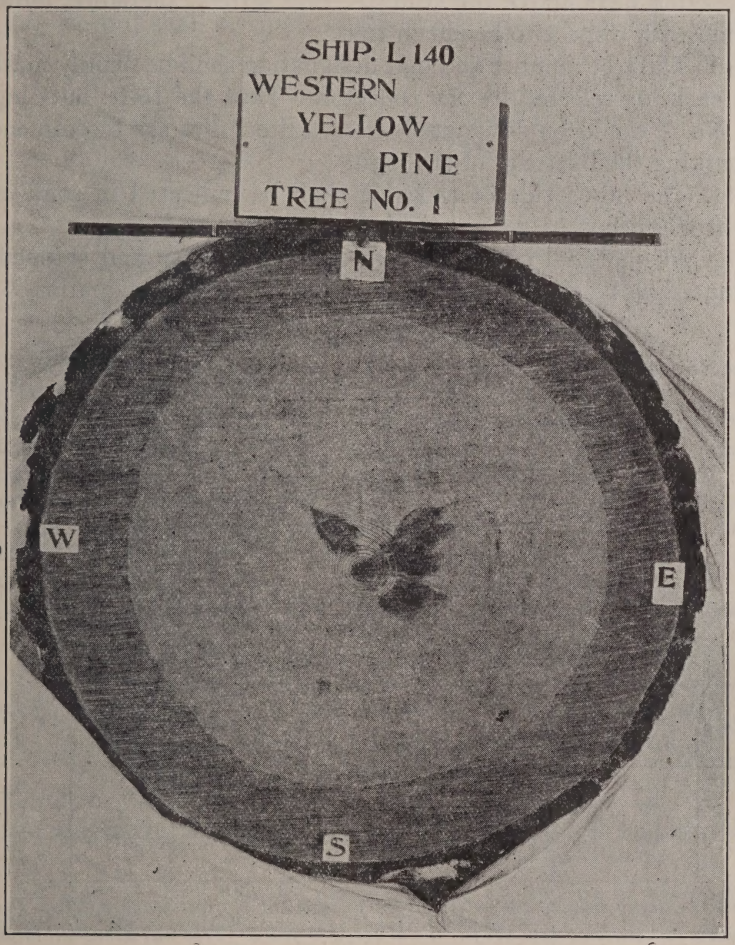

FIG. 1.—Section of Log Selected for Test Material.

7. When the trees are relatively small in diameter, material in addition to that provided for in Sections 5 and 6 shall be collected, whenever possible, to insure a sufficient number of tests to give a good average for the species. **Selection from Small Trees.**

(B) *Field Marking.*

Tree Designation.

8. Each tree shall be given an arabic number, the numbering in any given shipment to be consecutive for trees of a given species.

Bolt Designation.

9. Each 4 ft. of length of a tree or log shall be considered a "bolt." Bolts shall be designated by small letters, beginning with *a* for the 4-ft. section next above the stump. Bolt letters, therefore, indicate position with respect to height in tree.

Manner of Marking.

10. The tree number and bolt designation shall be plainly marked upon each log selected by the collector. Thus the 16-ft. butt log of Tree No. 2 would be designated 2*abcd*. Steel dies are recommended for marking the butt end of the logs.

Cardinal Point Indicated.

11. The north side of each log shall be indicated in some convenient manner.

Shipment Number.

12. All material collected from a given locality and shipped at one time shall be given a shipment number or other designation.

TABLE I.—SHIPMENT DESCRIPTION FIELD NOTES.

Project No., *124* Shipment No., *323*
Tree No., *10* Species, *Lodgepole pine*
Locality cut: State, *Montana* County, *Jefferson*
Township, *6 N., R. 7 W., M. P. M.* Date cut, *Oct. 10, 1914*
Slope, *Moderate* Absolute elevation, *6600 ft.* Aspect, *southeasterly*
Undergrowth, *None* Soil, *Deep, fresh-decomposed granite*
Height, *73.9 ft.* Seedling or sprout, *Seedling* Age, *140*
Crown, *Length 25 ft., width 12 ft.* D.B.H. *11.1 in.*
Stem, *Sound, smooth, clear, length 20 ft.* Date sawed, *Oct. 10, 1914*
How and when transported from woods, *By sleigh and wagon, Oct. 13 to 15, 1914.*

LIST OF SPECIMENS SAWED.

Log No.	Bolts.	Length, ft.	Diameter Inside Bark.		Volume.	Feetage, b. m.	Weight, Oct. 15, lb.
			Butt, in.	Top, in.			
1	bcd	12.1	11	9.6	..	..	336
2	efg	12.1	9.6	8.9	..	..	289
3	hij	12.1	8.9	7.7	..	..	242
4	klm	12.1	7.7	6.1	..	..	176
5	nop	12.1	6.1	3.1	..	..	95

REMARKS: *Stump height, 1.0 ft. Butt off on account of frost crack 3.0 ft. Length of top above last section taken, 9.4 ft. Total age, 140 years. Tree was dominant throughout life. Rate of growth, regular and good average for Site I. Lower slope type, although of more than average height for the age and diameter found on poorer sites.*

(C) *Field Descriptions.*

Field Description.

13. (*a*) Complete field notes describing the material shall be fully and carefully made by the collector. These notes shall in general, supply data as outlined in Table I.

(*b*) Photographs of the standing trees selected shall be taken.

(*D*) *Preparation for Shipment.*

14. (*a*) The bark will be left on each log, and care shall be taken to keep the bark intact. The ends of the logs shall be carefully painted to retard or prevent end drying and end checking. **Preparation for Shipment.**

(*b*) Record shall be made of the shipment routing, bill of lading,

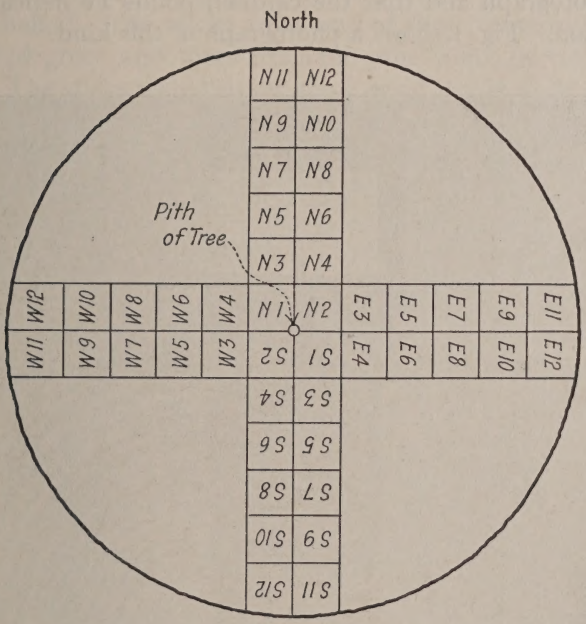

FIG. 2.—Sketch Showing Method of Cutting up the Bolt and Marking the Sticks.

kind of shipment, date of shipment, and condition of material when shipped.

(*c*) Record shall also be made of date of receipt of shipment at destination, its condition and method of storage.

DISPOSITION

(*A*) *Storage of Logs at Destination.*

15. Material shall not be kept in the bolt or log form long enough to permit damage by checks, decay, stains, or insect attack. The **Storage of Logs.**

logs shall be piled on skids, free from contact with the soil, and shall not be stored where subjected to artificial heat. In addition they should preferably be protected from the sun.

(*B*) *Photographing, Sawing and Final Marking.*

Photographing Ends of Bolts.

16. The top end of each *d* or *c-d* bolt shall be photographed. It is suggested that a rule be so placed on the log as to indicate the scale of the photograph and that the cardinal points be indicated on the cross-section. Fig. 1 shows a photograph of this kind.

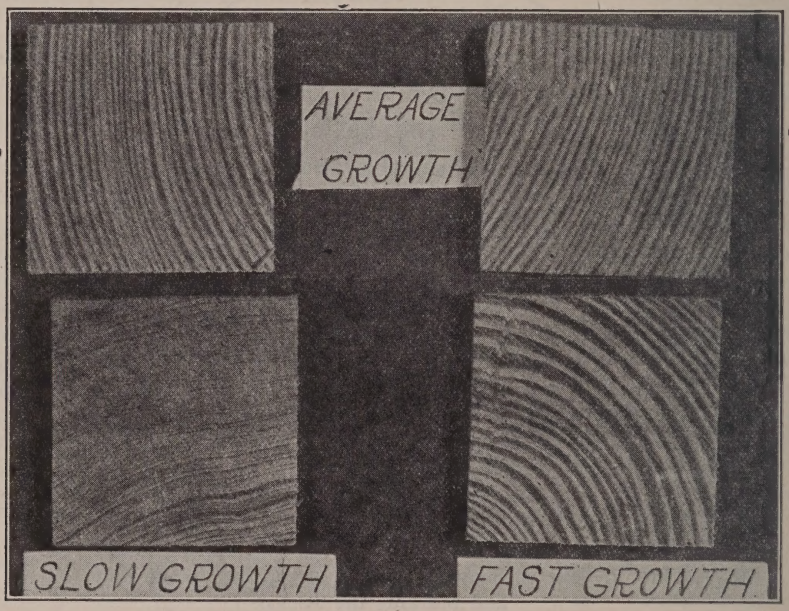

FIG. 3.—Cross-Sections of Bending Specimens Showing Different Rates of Growth of Longleaf Pine (2 by 2-in. Specimens).

Sawing of Bolts.

17. (*a*) All bolts shall be marked on the top end into $2\frac{1}{2}$ by $2\frac{1}{2}$-in. squares as shown in Fig. 2, and sawed into nominal $2\frac{1}{2}$ by $2\frac{1}{2}$-in. sticks. The letters *N*, *E*, *S*, and *W* indicate the cardinal points.

(*b*) When flitches are substituted for bolts (Section 6), the same general marking and numbering scheme of Paragraph (*a*) shall be followed in so far as it is applicable.

Marking of Test Sticks.

18. All test sticks shall bear the shipment number, the tree number, stick number, and bolt designation, to be known respectively

as Shipment No., Piece No., Stick No., and mark. Thus, 400-1-*N*4*d* represents Stick *N*4 of Bolt *d*, Tree 1, Shipment 400.

(*C*) *Matching for Air-dry Tests.*

Composition Bolts.

19. In the tests made to determine the effect of air-drying on strength, the sticks from the *c* bolt shall be interchanged with those of the *d* bolt of the same tree to form two composition bolts, each composition bolt being complete and being made of equal portions of *c* and *d* bolts.[1] The sticks from one of these composition bolts shall be tested green and those from the other shall be tested after air drying. The sticks tested either green or air dry shall be regarded as if they were from the same bolt.

Schedule for Forming Composition Bolts.

20. The division of green and air-dry sticks shall be made according to the following schedule in which the numbers refer to stick numbers:

Composition Bolt to be Tested Green.—

Lower bolt *c*	1	4, 5	8, 9	
Upper bolt *d*	2, 3	6, 7		10, etc.

Composition Bolt to be Air Dried and Tested.—

Lower bolt *c*	2, 3	6, 7		10, etc.
Upper bolt *d*	1	4, 5	8, 9	

(*D*) *Disposition of Sticks.*

Green Material.

21. The sticks ($2\frac{1}{2}$ by $2\frac{1}{2}$ in. by 4 ft.) to be tested green shall be kept in an unseasoned condition while awaiting preparation for test by being stored in a framed pit or other suitable container, where they shall be close piled and covered with damp sawdust or in some other suitable manner. As material is required for test, it shall be removed from this pit, or container, surfaced on all four sides to 2-in. by 2-in. in cross-section, sawed to test size and kept covered with a damp cloth, in an air-tight container at a temperature of about 70° F. (as nearly as practicable) until the time of test. Care shall be taken to avoid as much as possible the storage of green material in any form. Sticks to be tested in a green condition should usually not be sawed from the log form in quantities greater than is required to meet the

[1] The 8-ft. section (*c* and *d* bolts) from each of the two trees provided for under Section 5 (*b*) is intended for tests to determine the effect of air drying on strength.

testing demands for from a few days to not more than two weeks, depending on the prevailing conditions.

Air-dry Material.

22. (*a*) The ends of the sticks to be air dried ($2\frac{1}{2}$ by $2\frac{1}{2}$ in. by 4 ft.) shall be dipped in melted paraffin or other substance suitable to retard checking. The material shall be piled so as to have a space of at least $\frac{1}{2}$ in. on each side of each stick to permit circulation of air. The material shall be stored in a place allowing free access of air, but protected from sunshine, rain, snow, and moisture from the ground. The sticks in drying shall not be subjected to artificial heat.

(*b*) All of the sticks from each composite bolt to be air dried shall be weighed when stored and at sufficiently frequent intervals thereafter to get accurate data on the progress of seasoning. No

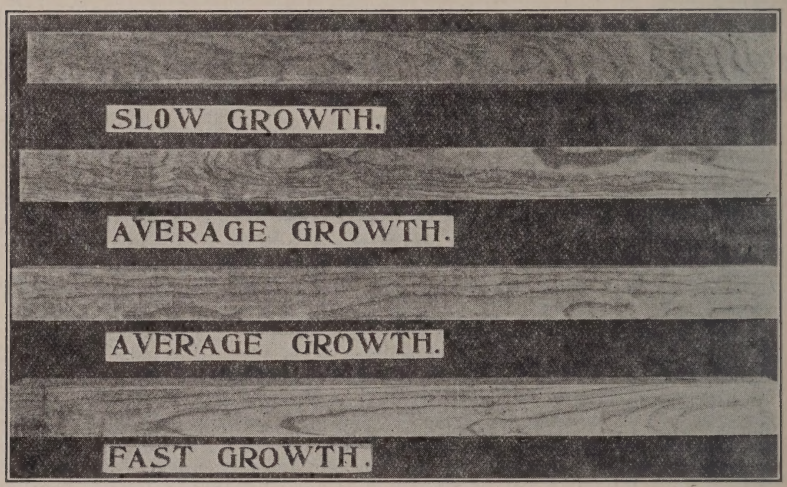

FIG. 4.—Tangential Surfaces of Bending Specimens of Different Rates of Growth of Jeffrey Pine (2 by 2 by 30-in. Specimens).

material shall be considered thoroughly air dried and properly conditioned for testing until practically constant weight has been reached. (Wood absorbs and gives off moisture with changing atmospheric conditions, consequently it never comes to absolutely constant weight.)

(*c*) When the material has reached equilibrium, moisture sections 1 in. in length shall be taken from about 10 per cent of the sticks to determine the actual moisture content. These moisture specimens shall be cut not less than 1 ft. from the ends of the sticks, and in such a way as to prevent any appreciable loss of material for testing. When thoroughly air dry, the material shall be surfaced on four sides to 2 in. by 2 in. in cross-section and tested.

TESTING

ORDER, SELECTION, AND NUMBER

(*A*) *Order.*

Order of Tests.

23. The order of tests in all cases shall be such as to eliminate as far as possible from the comparisons the effect of changes in the specimen due to such factors as storage and weather conditions.

(*B*) *Selection.*

Preference in Selecting Specimens.

24. In case the material from a given bolt should be insufficient to furnish all the test specimens hereinafter required, the preferential order of mechanical tests to be used in selecting specimens shall be as follows: Static bending, compression parallel to grain, impact bending, compression perpendicular to grain, hardness, shear parallel to grain, tension perpendicular to grain, cleavage, and tension parallel to grain.

Test Pieces from Bending Specimens After Failure.

25. In many cases where a shortage of material exists, certain test specimens may be taken from the uninjured portion of the static and impact bending specimens remaining after test, provided proper care is used in the selection.

Quality of Test Material.

26. Only clear straight-grained material, free from decay and other defects, shall be used for the tests. However, small knots and other similar defects may be admitted in such specimens as static bending when their location is such that it is certain they will not in any way influence the failure or otherwise affect the strength of the specimen.

(*C*) *Number of Tests for Each Bolt.*

Static Bending.

27. One static bending specimen shall be taken from each pair of sticks. A pair consists of two adjacent sticks equi-distant from the pith, as *W*3 and *W*4, Fig. 2. In the composite bolts tested to afford a comparison of the strength of green and air-dry material, the pair of sticks shall be constituted as above, except that the sticks in this case will be from different bolts. Thus *W*3*d* and *W*4*c* constitute one pair of sticks to be tested green, and *W*3*c* and *W*4*d* the corresponding pair to be tested air dry (Section 20).

Compression Parallel to Grain.

28. One compression-parallel-to-grain specimen shall be taken from each stick. Load-compression curves shall be taken on 20 per cent of the specimens.

Impact Bending.

29. Eight impact-bending specimens shall be taken from each bolt, selection being made from the sticks remaining after obtaining the static bending tests. Two of the specimens shall be selected from

near the pith, two from near the periphery, and four which are representative of the cross-section.

Compression Perpendicular to Grain.

30. One compression-perpendicular-to-grain specimen shall be taken from each of 50 per cent of the sticks selected for static bending.

Hardness.

31. One hardness specimen shall be taken from each of the other 50 per cent of the static-bending sticks.

Shear Parallel to Grain.

32. Eight shear-parallel-to-grain specimens shall be selected from the unused portion or ends of four sticks from which bending specimens have been selected. Two specimens shall be taken from near the pith, two from near the periphery, and four that are representative of the average growth of the cross-section of the bolt. One of each pair of specimens from the same stick shall be tested in radial shear (surface of failure radial) and the other in tangential shear (surface of failure tangential).

Tension Perpendicular to Grain.

33. Eight tension-perpendicular-to-grain specimens shall be selected from four sticks in a manner similar to that for shear (Section 32). One of each pair of specimens from the same stick shall be tested in radial tension (surface of failure radial) and the other in tangential tension (surface of failure tangential).

Cleavage Perpendicular to Grain.

34. Eight cleavage specimens shall be selected from four sticks in a manner similar to that for shear (Section 32). One of each pair of specimens from the same stick shall be tested in radial cleavage (surface of failure radial) and the other in tangential cleavage (surface of failure tangential).

Tension Parallel to Grain.

35. Four tension-parallel-to-grain specimens shall be chosen of which one shall be selected from near the pith, one from near the periphery and two which are representative of the cross-section.

Specific Gravity and Shrinkage in Volume.

36. Six specific gravity and shrinkage-in-volume specimens shall be selected from the unused portion of bending or tension-parallel-to-grain sticks, selected so as to give one from near the pith, one from near the periphery, and four that are representative of the average growth of the cross-section of the bolt. These specimens shall be selected only from the sticks to be tested in a green condition.

Radial Shrinkage.

37. Two radial shrinkage specimens shall be obtained from each *d* bolt. They shall be cut from the "sectors" or "quadrants" remaining after sawing (Fig. 2) or from disks cut from near the end of the bolt. When a disk is used, care must be taken to see that it is green, and has not been affected by shrinking and checking which is common near the end of the bolt. The specimens shall not be surfaced. Radial shrinkage specimens shall be cut with their width in the radial direction. One shall be taken from the heartwood, and the other from near the periphery. When possible, the second shall consist entirely of sapwood.

38. Two tangential shrinkage specimens shall be obtained from each *d* bolt. They shall be selected at the same time and in a manner similar to radial-shrinkage specimens (Section 37), except the width shall be in a tangential direction. The specimens shall not be surfaced. One shall be taken from the heartwood, the other from near the periphery and when possible shall consist entirely of sapwood. The heartwood and the sapwood specimens shall be taken adjacent to the respective specimens selected for radial shrinkage. **Tangential Shrinkage.**

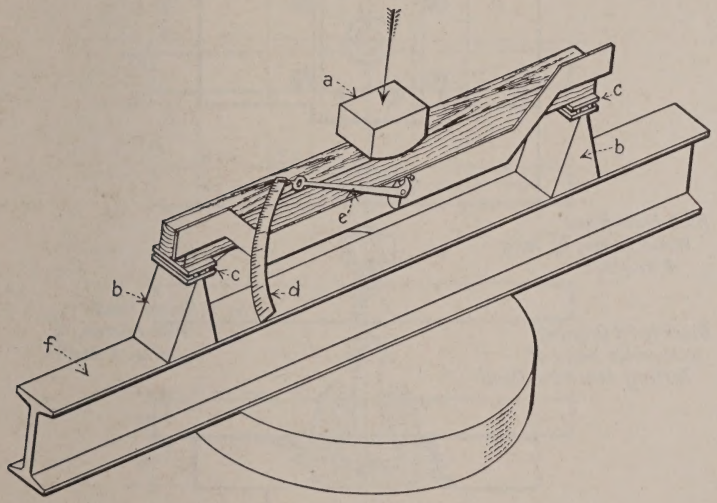

FIG. 5.—Diagrammatic Sketch of Method of Conducting Static Bending Test.

GENERAL INSTRUCTIONS

(*A*) *Photographs of Sticks.*

39. Four of the bending sticks from each species shall be selected for photographing, as follows: 2 average growth, 1 fast growth, and 1 slow growth. These sticks shall be photographed in cross-section and on the radial and tangential surfaces. Fig. 3 is a typical photograph of the cross-section of test specimens and Fig. 4 of the tangential surface of bending specimens. **Sticks to be Photographed.**

(*B*) *Control of Moisture Content.*

40. As previously specified (Section 22) sticks for test in the air-dry condition shall be brought to practically constant weight **Control of Moisture Content.**

before test. Tests shall then be carried out in such a manner that large changes in moisture content will not occur. (For instance, in ordinary heated rooms the relative humidity is usually below 30 per cent. If wood which has been dried to practically constant

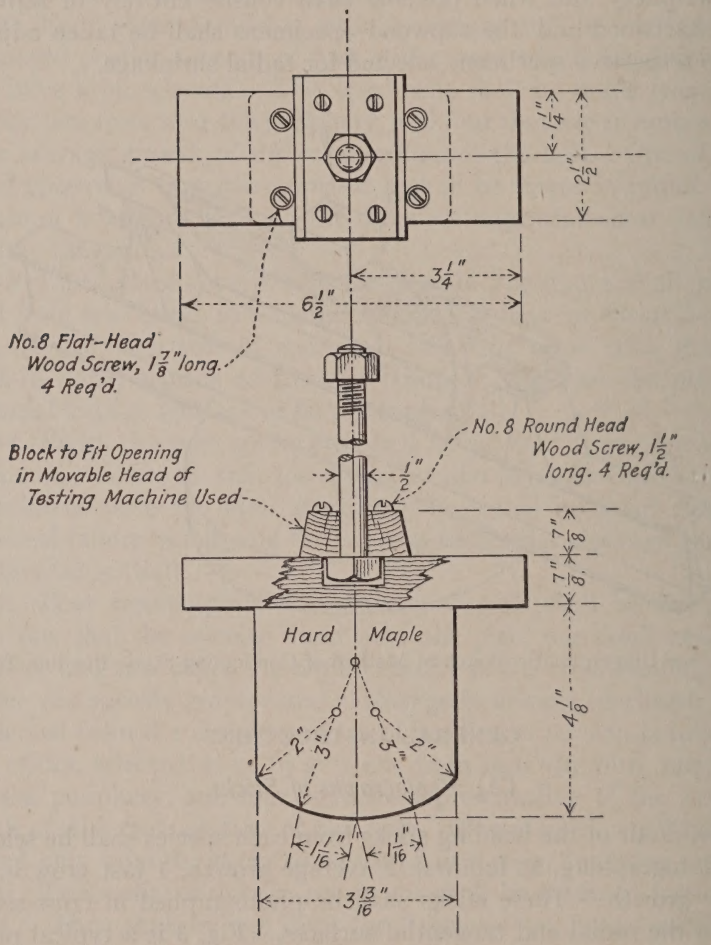

FIG. 6.—Details of Bearing Block for Bending Tests.

weight under the condition specified by Section 22 is brought into such a room, a considerable and rather rapid drying out will occur.) To prevent such changes, the testing room and rooms for the preparation of test specimens should preferably have some means of humidity control.

(C) Record of Heartwood and Sapwood.

41. The estimated proportion of sapwood present shall be recorded for each test specimen. **Proportion of Sapwood.**

PROCEDURE

(A) Static Bending.

42. The static bending tests shall be made on nominal 2 by 2 **Size of Specimens.**

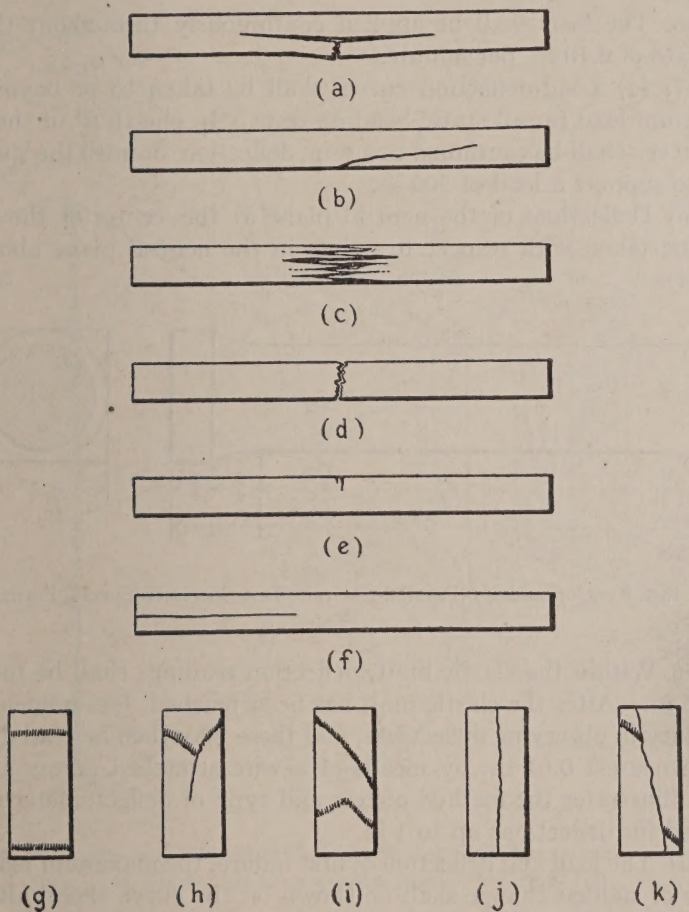

FIG. 7.—Types of Failures: *a* to *f*, Bending; *g* to *k*, Compression Parallel to Grain.

by 30-in. specimens. The actual height and width at the center, and the length shall be measured.

43. Center loading and a span length of 28 in. shall be used. Both supporting knife edges shall be provided with bearing plates and rollers having a total thickness of approximately $1\frac{1}{4}$ in. in order to **Loading Span and Supports.**

bring the neutral plane of the test specimen about $2\frac{1}{4}$ in. from the knife edges (Fig. 5.)

Bearing Block.

44. A bearing block of the form and size of that shown in Fig. 6 shall be used for applying the load.

Placement of Growth Rings.

45. The specimen shall be placed so that the load will be applied through the bearing block to the tangential (flat-sawn) surface nearest the pith.

Rate of Application of Load.

46. The load shall be applied continuously throughout the test at a rate of 0.10 in. per minute.

Load-Deflection Curves.

47. (*a*) Load-deflection curves shall be taken to or beyond the maximum load for all static bending tests. In one-third of the tests, the curves shall be continued to a 6-in. deflection, or until the specimen fails to support a load of 200 lb.

(*b*) Deflections of the neutral plane at the center of the length shall be taken with respect to points in the neutral plane above the supports.

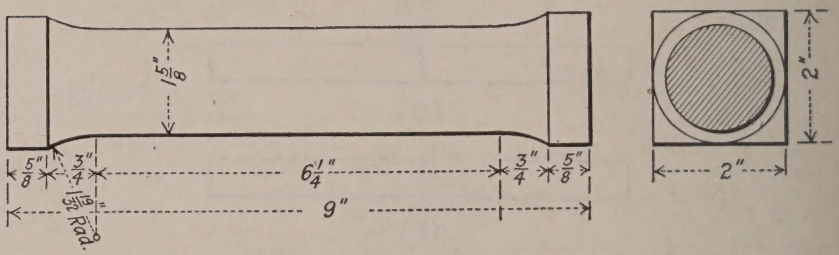

FIG. 8.—Compression-Parallel-to-Grain Test Specimen Special Form.

(*c*) Within the elastic limit, deflection readings shall be taken to 0.0025 in. After the elastic limit has been reached, less refinement is necessary in observing deflections, and these may then be read directly to the nearest 0.01 in. by means of a wire stretched across a scale. Fig. 5 illustrates the method of test and type of deflectometer recommended for deflections up to 1 in.

(*d*) The load and deflection of first failure, the maximum load, and points of sudden change shall be shown on the curve sheet[1] although they may not occur at one of the regular load or deflection increments.

Description of Failures.

48. Bending failures shall be classified according to the appearance of the fractured surface and according to the manner in which the failure develops. The fractured surfaces may be roughly divided into brash and fibrous, the term "brash" indicating an abrupt failure

[1] See Fig. 1 of the Appendix for a sample static bending data sheet form. Fig. 2 of the Appendix shows a sample computation data card.

and the term "fibrous" indicating a fracture showing splinters. The test failures shall be classified and described as follows:

Simple tension (see Fig. 7*a*);

Cross grain[1] tension (see Fig. 7*b*);

The presence of cross grain having a slope which deviates more than 1 in 20 from the longitudinal edges of the specimen shall be cause for culling the test.

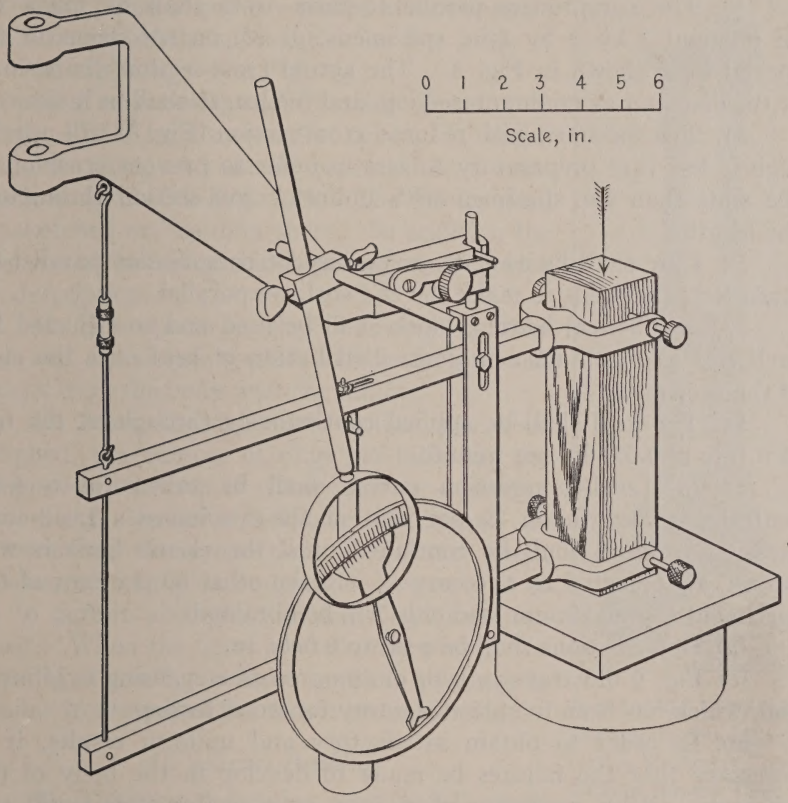

FIG. 9.—Diagrammatic Sketch of U. S. Forest Service Compressometer and Method of Conducting Compression-Parallel-to-Grain Test.

Splintering tension (see Fig. 7*c*);

Brash tension (see Fig. 7*d*);

Compression (see Fig. 7*e*);

Horizontal shear (see Fig. 7*f*).

[1] The term "cross grain" shall be considered to include all deviations of grain from the direction of the longitudinal axis or longitudinal edges of the specimen. It should be noted that spiral grain may be present even to a serious extent without being evident from a casual observation.

In case two or more kinds of failures develop, all shall be described in the order of their occurrence; thus, compression followed by splintering tension. The failure shall be sketched on the data sheet.

Weight and Moisture Content.

49. The specimen shall be weighed after test and a moisture section 1 in. in length shall be cut near the failure.

(*B*) *Compression Parallel to Grain.*

Size of Specimens.

50. The compression-parallel-to-grain tests shall be made (1) on nominal 2 by 2 by 8-in. specimens, or (2) on specimens of the special form shown in Fig. 8. The actual cross-section dimensions, or the diameter at minimum section, and the length shall be measured.

Advantage of Special Form.

51. The specimen with reduced cross-section (Fig. 8) will usually require less care preparatory to test in order to prevent crushing at the ends than the specimen with uniform cross-section throughout the length.

End Surfaces Parallel.

52. Care shall be used in preparing the compression-parallel-to-grain test specimens to make the end surfaces parallel.

Spherical Bearing.

53. A spherical bearing block shall be used and so adjusted for each test as to obtain a uniform distribution of load over the ends of the specimen.

Rate of Application of Load.

54. The load shall be applied continuously throughout the test at a rate of 0.024 in. per minute.

Load-Compression Curves.

55. (*a*) Load-compression curves shall be taken for a 6-in. central gage length on 20 per cent of the specimens. Load-compression readings shall be continued until the elastic limit is well passed, as indicated by the curve.[1] On the other 80 per cent of the specimens the maximum load only will be obtained.

(*b*) Deformations shall be read to 0.0005 in.

(*c*) Fig. 9 illustrates a type of compressometer, using a Johnson dial, which has been found satisfactory for wood testing.

Position of Test Failures.

56. In order to obtain satisfactory and uniform results, it is necessary that the failures be made to develop in the body of the specimen. With specimens of uniform cross-section, this result can best be obtained when the ends are at a very slightly lower moisture content than the body. With green material it will usually suffice to close-pile the specimens, cover the body with a damp or wet cloth and expose the ends for a short time. For air-dry material, it may sometimes be advisable to pile the specimens in a similar manner and place them in a desiccator should the failures in test indicate that a slight end drying is necessary.

[1] See Fig. 4 of the Appendix for a sample compression-parallel-to-grain data sheet form. Fig. 3 of the Appendix shows a sample computation data card.

57. Compression failures shall be classified and described according to their appearance, as follows: Description of Failures.

(*a*) *Crushing.*—This term shall be used when the plane of rupture is approximately horizontal. See Fig. 7(*g*).

(*b*) *Wedge Split.*—The direction of the split, that is whether radial or tangential, should be noted. See Fig. 7(*h*).

(*c*) *Shearing.*—This term shall be used when the plane of rupture makes an acute angle with the axis of the specimen. See Fig. 7 (*i*).

(*d*) *Splitting.*—See Fig. 7(*j*).

(*e*) *Compression and Shearing Parallel to Grain.*—This failure usually occurs in cross-grained pieces, and should be culled as a compression test. See Fig. 7(*k*). The failure shall be sketched on the data sheet. In addition the location of the failure shall be recorded along with its description.

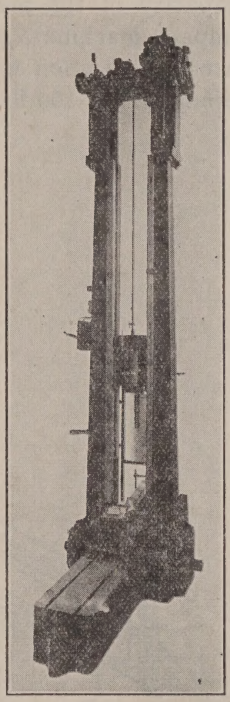

FIG. 10.—Hatt-Turner Impact Machine, Illustrating Method of Conducting Impact Bending Test.

58. The specimen shall be weighed after test and a moisture section 1 in. in length shall be cut from the body near the failure. Weight and Moisture Content.

59. After the moisture section has been weighed, the number of rings per inch and the proportion of summer wood shall be measured over a representative inch of cross-section. In determining the proportion of summer wood, it is essential that the end surface be prepared so as to permit accurate summer wood measurement. When the fibers are broomed over at the ends from sawing, a light sanding, planing, or similar treatment of the ends is recommended. Ring and Summer Wood Measurement.

(*C*) *Impact Bending.*

60. The impact bending tests shall be made on nominal 2 by 2 by 30-in. specimens. The actual height and width at the center and the length shall be measured. Size of Specimens.

61. Center loading and a span length of 28 in. shall be used. Loading and Span.

62. A metal tup of curvature corresponding to the bearing block shown in Fig. 6 shall be used in applying the load. Bearing Block.

63. The specimen shall be placed so that the load will be applied through the bearing block to the tangential or flat-sawn surface nearest the pith. Placement of Growth Rings.

Method of Test.

64. The tests shall be made by increment drops in a Hatt-Turner impact or similar machine. See Fig. 10. The first drop shall be 1 in., which shall be increased by 1-in. increments until a height of 10 in. is reached. A 2-in. increment shall then be used until complete failure occurs or until a 6-in. deflection is reached.

Weight of Hammer.

65. A 50-lb. hammer shall be used when, with drops up to the capacity of the machine (about 68 in. for the small Hatt-Turner impact machine), it is practically certain that complete failure or a 6-in. deflection will result for all specimens of a species. For all other cases a 100-lb. hammer shall be used.

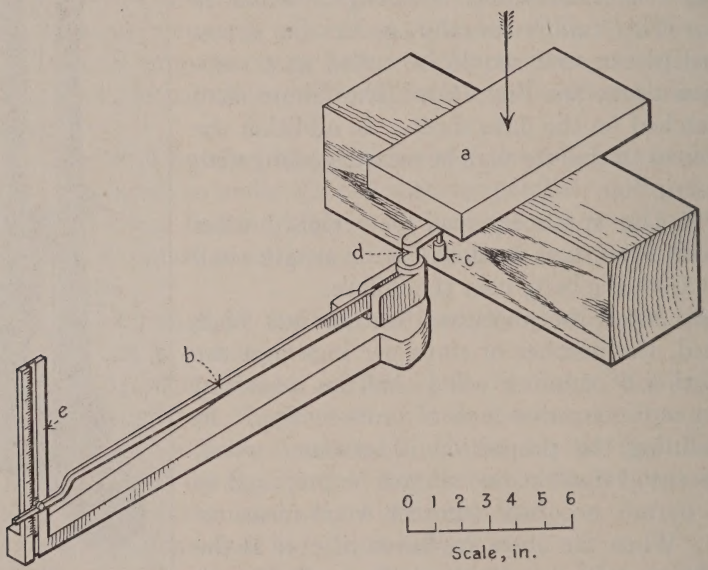

FIG. 11.—Diagrammatic Sketch of Method of Conducting Compression-Perpendicular-to-Grain Test.

Deflection Records.

66. Graphical drum records,[1] giving the deflection for each drop and the set, if any, shall be made until first failure occurs. This record shall also afford data from which the exact height of drop can be scaled for at least the first four falls.

Drop Causing Failure.

67. The height of drop causing complete failure, or a 6-in. deflection, shall be observed for each specimen.

Description of Failure.

68. The failure shall be sketched on the data sheet[2] and described in accordance with the directions for static bending under Section 48.

[1] See Fig. 5 of the Appendix for a sample drum record.

[2] See Fig. 7 of the Appendix for a sample impact bending data sheet form. Fig. 6 of the Appendix shows a sample computation data card.

69. The specimen shall be weighed after the test and a moisture section 1 in. in length shall be cut near the failure. **Weight and Moisture Content.**

(D) Compression Perpendicular to Grain.

70. The compression-perpendicular-to-grain tests shall be made on nominal 2 by 2 by 6-in. specimens. The actual height, width, and length shall be measured. **Size of Specimens.**

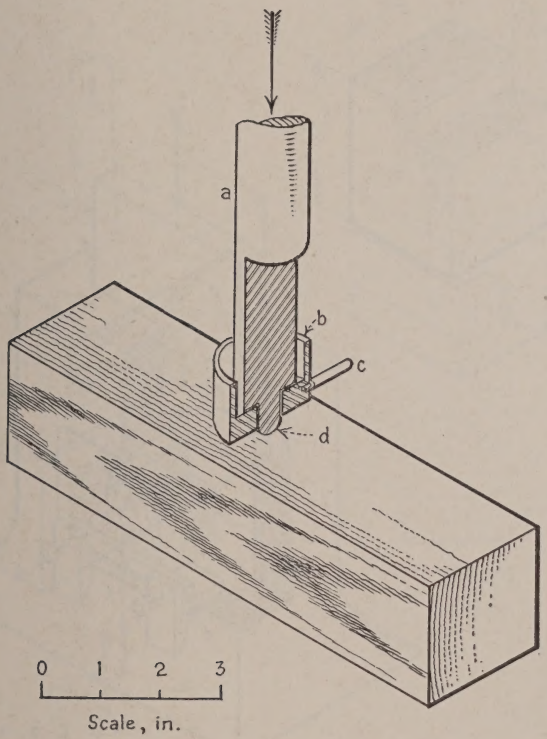

FIG. 12.—Diagrammatic Sketch of Method of Conducting Hardness Test.

71. The load shall be applied through a metal bearing plate 2 in. in width, placed across the upper surface of the specimen at equal distances from the ends and at right angles to the length. (See Fig. 11.) **Loading.**

72. The specimen shall be placed so that the load will be applied through the bearing plate to a radial (quarter-sawn) surface. **Placement of Growth Rings.**

73. The load shall be applied continuously throughout the test at a rate of 0.024 in. per minute. **Rate of Application of Load.**

Load-Compression Curves.

74. (*a*) Load-compression curves[1] shall be taken for all specimens up to 0.1-in. compression, after which the test will be discontinued.

(*b*) Deflection readings shall be taken to 0.001 in.

Weight and Moisture Content.

75. The specimen shall be weighed after test and a moisture section 1 in. in length shall be cut adjacent to the part under load.

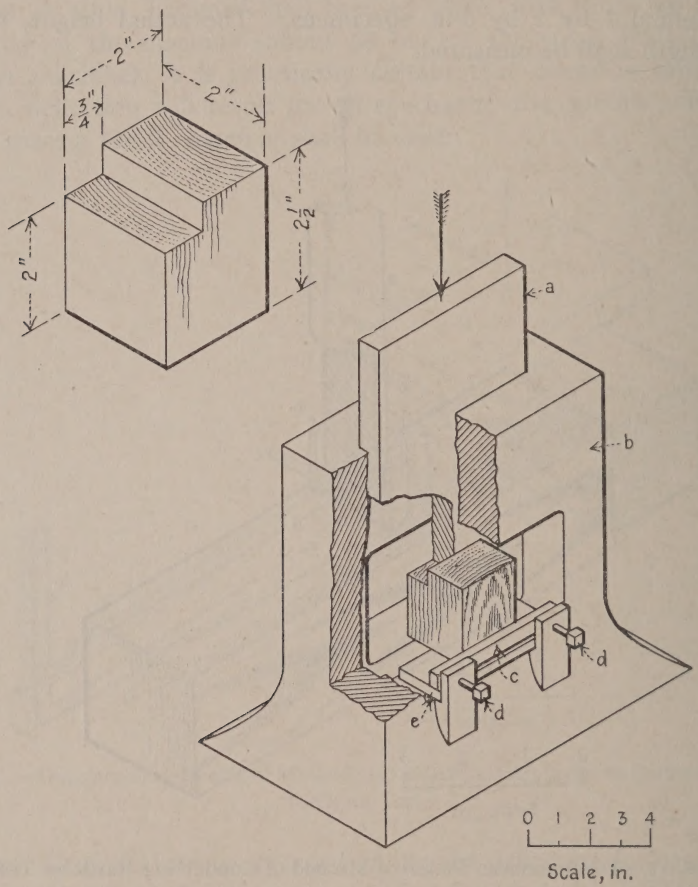

FIG. 13.—Diagrammatic Sketch of Method of Conducting Shear-Parallel-to-Grain Test, with Details of Test Specimen.

(*E*) *Hardness.*

Size of Specimens.

76. The hardness tests shall be made on nominal 2 by 2 by 6-in. specimens. The actual cross-section dimensions and length shall be measured.

[1] See Fig. 8 of the Appendix for a sample compression-perpendicular-to-grain data sheet form. Fig. 9 of the Appendix shows a sample computation data card.

77. The modified ball test with a "ball" 0.444 in. in diameter shall be used for determining hardness. (See Fig. 12.) The load at which the "ball" has penetrated to one-half its diameter as indicated by the tightening of the collar against the specimen, shall be observed. Method of Test.

78. Two penetrations shall be made on a tangential surface, two on a radial surface, and two on each end. The choice between Number of Penetrations.

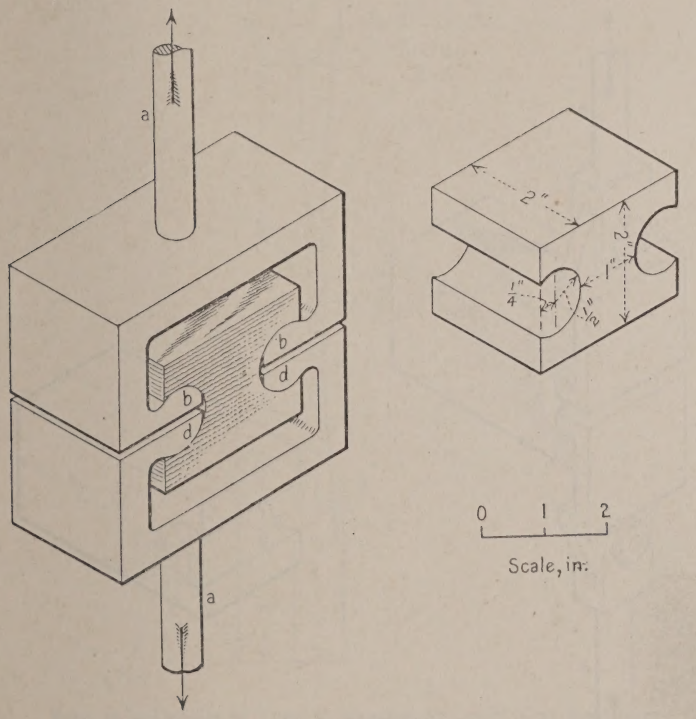

FIG. 14.—Diagrammatic Sketch of Method of Conducting Tension-Perpendicular-to-Grain Test, with Details of Test Specimen.

the two radial and between the two tangential surfaces shall be such as to give a fair average of the piece. The penetrations shall be far enough from the edge to prevent splitting or chipping.[1]

79. The load shall be applied continuously throughout the test at a rate of 0.25 in. per minute. Rate of Application of Load.

80. The specimen shall be weighed after test and a moisture section 1 in. in length shall be cut. Weight and Moisture Content.

[1] See Fig. 10 of the Appendix for a sample data and computation card.

(F) Shear Parallel to Grain.

Size of Specimens. 81. The shear-parallel-to-grain tests shall be made on nominal 2 by 2 by $2\frac{1}{2}$-in. specimens notched as illustrated in Fig. 13 to produce failure on a 2 by 2-in. surface. The actual dimensions of the shearing surface shall be measured.

Method of Test. 82. A shear tool similar to that illustrated in Fig. 13, providing a $\frac{1}{8}$-in. offset between the inner edge of the supporting surface and

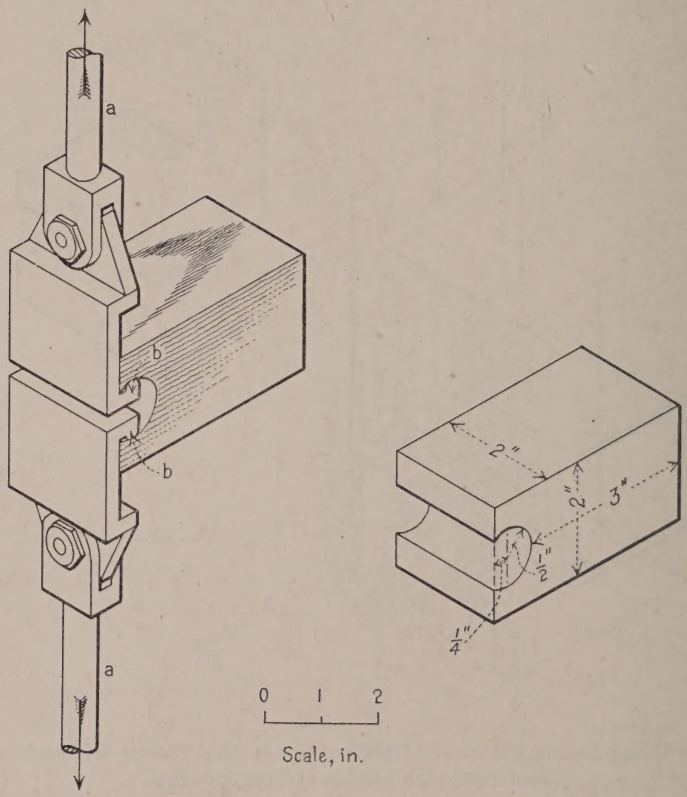

FIG. 15.—Diagrammatic Sketch of Method of Conducting Cleavage Test, with Details of Test Specimen.

the plane along which the failure occurs, shall be used. The load shall be applied and the specimen supported on end-grain surfaces. Care shall be taken in placing the specimen in the shear tool to see that the cross bar is adjusted so that the edges of the specimen are vertical and the end rests evenly on the support over the contact area. The maximum load only will be observed.

83. The load shall be applied continuously throughout the test at a rate of 0.015 in. per minute. **Rate of Application of Load.**

84. The failure shall be sketched on the data card.[1] In all cases where the failure at the base of the specimen extends back onto the supporting surface, the test shall be culled. **Test Failures.**

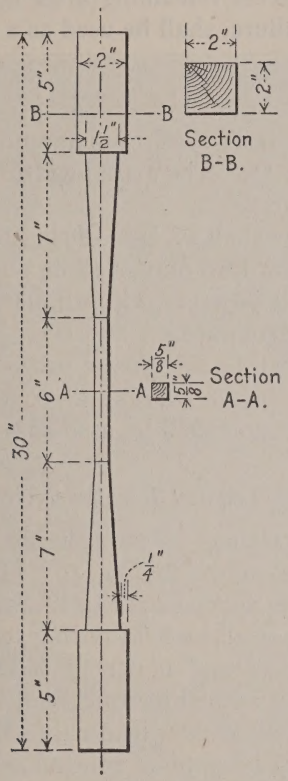

FIG. 16.—Details of Tension-Parallel-to-Grain Test Specimen.

85. The portion of the test piece which is sheared off shall be used as a moisture section. **Moisture Content.**

(G) *Tension Perpendicular to Grain.*

86. The tension-perpendicular-to-grain tests shall be made on specimens of the nominal size and shape shown in Fig. 14. The actual width and length at minimum sections shall be measured. **Size of Specimens.**

[1]See Fig. 11 of the Appendix for a sample tangential-shear-parallel-to-grain data sheet form.

Method of Test.

87. The specimens shall be held during test in grips as shown in Fig. 14. The maximum load only shall be observed.

Rate of Application of Load.

88. The load shall be applied continuously throughout the test at a rate of 0.25 in. per minute.

Sketch of Failure.

89. The failure shall be sketched on the data card.[1]

Moisture Content.

90. One of the pieces remaining after failure, or a section split along the surface of failure, shall be used as a moisture specimen.

(*H*) *Cleavage.*

Size of Specimens.

91. The cleavage tests shall be made on specimens of the form and size shown in Fig. 15. The actual width and length at minimum section shall be measured.

Method of Test.

92. The specimens shall be held during test in grips as shown in Fig. 15. The maximum load only shall be observed.

Rate of Operation.

93. The load shall be applied continuously throughout the test at a rate of 0.25 in. per minute.

Sketch of Failure.

94. The failure shall be sketched on the data card.[2]

Moisture Content.

95. One of the pieces remaining after failure, or a section split along the surface of failure, shall be used as a moisture specimen.

(*I*) *Tension Parallel to Grain.*

Size of Specimens.

96. The tension-parallel-to-grain tests shall be made on specimens of the size and shape shown in Fig. 16. The actual cross-section dimensions at minimum section shall be measured.

Method of Test.

97. The specimen shall be held during test by plates notched to fit around the test piece and under the 0.25-in. shoulder provided for that purpose. A spherical bearing shall be used on at least one end of the specimen. Maximum load only will be observed.

Rate of Application of Load.

98. The load shall be applied continuously throughout the test at a rate of 0.05 in. per minute.

Sketch of Failure.

99. The failure shall be sketched on the data sheet.[3]

Moisture Content.

100. A moisture section about 3 in. in length shall be cut from the minimum section near the failure.

(*J*) *Specific Gravity and Shrinkage in Volume.*

Size of Specimens.

101 The specific gravity and shrinkage in volume tests shall be made on nominal 2 by 2 by 6-in. specimens. The actual cross-section dimensions and length shall be measured.

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[1] See Fig. 12 of the Appendix for a sample radial-tension-perpendicular-to-grain data sheet form.
[2] See Fig. 13 of the Appendix for a sample radial-cleavage data sheet form.
[3] See Fig. 14 of the Appendix for a sample tension-parallel-to-grain data sheet form.

102. (*a*) Both specific gravity and shrinkage-in-volume determinations shall be obtained on the same specimen. Determination Method.

(*b*) A carbon impression of the end of the green specimen shall be made on the back of the data card.[1] In like manner, a carbon impression of the same end shall be made after the specimen has been oven dried. (See Paragraph *e*.)

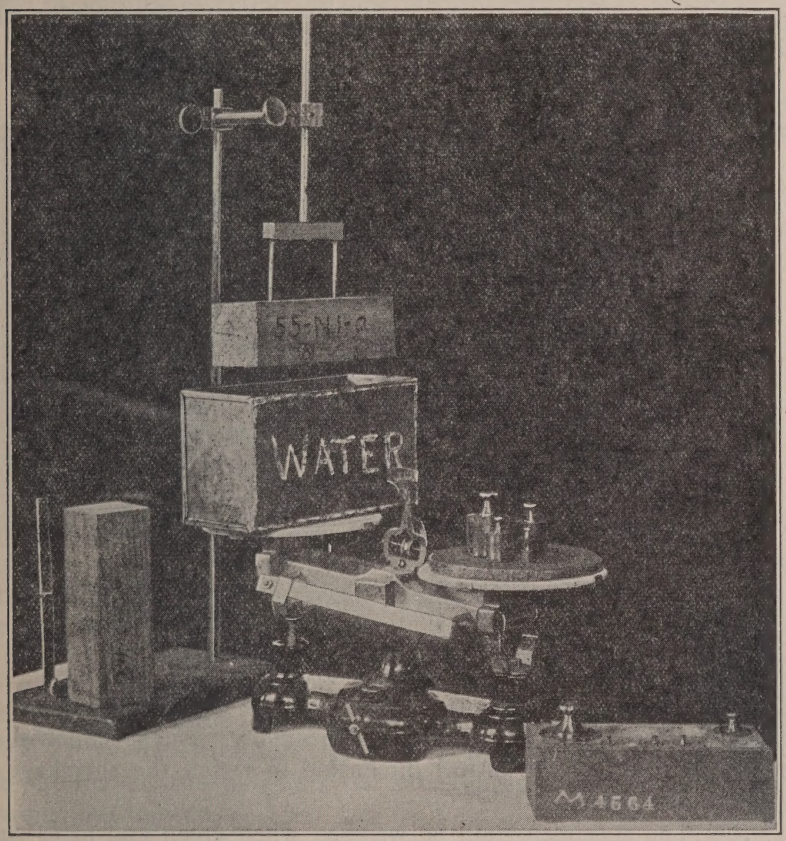


FIG. 17.—Method of Conducting Specific Gravity and Shrinkage-in-Volume Test.

(*c*) The specimen shall be weighed when green and the volume shall be determined by the immersion method.

(*d*) The green specimens after immersion shall be open-piled and allowed to air season under room conditions until approximately constant weight is attained.

[1] See Fig. 15 of the Appendix for a sample specific gravity and shrinkage-in-volume data card form.

(*e*) The specimens shall then be open-piled in an oven and dried at 100° C. until constant weight is reached.

(*f*) After oven drying, the specimens shall be weighed, and while still warm shall be immersed in a hot paraffin bath, care being taken to remove them quickly to insure a thin coating.

(*g*) The volume of the paraffin-coated specimen shall be determined by immersion as before.

(*h*) Fig. 17 illustrates the apparatus used in determining the specific gravity and shrinkage in volume.

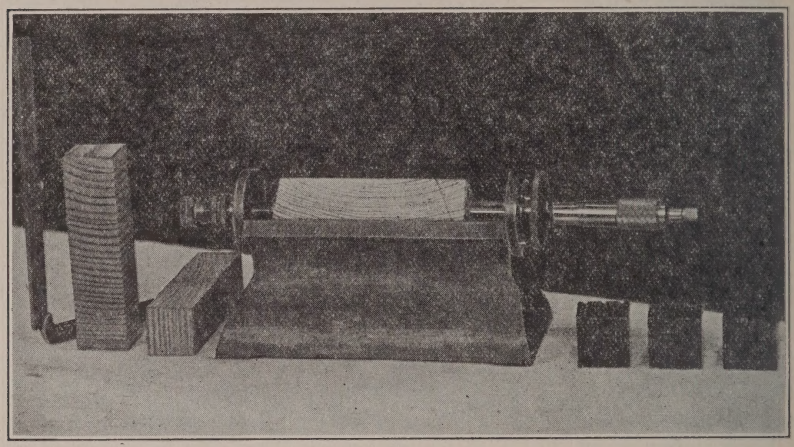


FIG. 18.—Method of Measuring Radial and Tangential Shrinkage.

(*K*) *Radial and Tangential Shrinkage.*

Size of Specimen. 103. The radial and tangential-shrinkage determinations shall be made on nominal 1 by 4 by 1-in specimens. The specimen shall be measured across the (4-in.) dimension in which the shrinkage is to be determined.

Weight. 104. The specimen shall be weighed when green and after subsequent oven drying.

Drying. 105. (*a*) The green specimens shall be open-piled and allowed to air season under room conditions until approximately constant weight is attained.

(*b*) The specimens shall then be open-piled in an oven and dried at 100° C. until constant weight is attained.

Final Measurement. 106. Final measurement shall be made on the oven-dry specimen.[1]

[1] See Fig. 16 of the Appendix for a sample radial and tangential-shrinkage data card form.

107. Figure 18 illustrates the method of making the radial and tangential-shrinkage measurements. An ordinary micrometer of required accuracy is suitable for this work.

(*L*) *Moisture Determination.*

Selection. 108. The sample for moisture determinations of each test specimen shall be selected as heretofore described for each test.

Weighing. 109. Immediately after obtaining the moisture sample, all loose splinters shall be removed and the sample shall be weighed.

Drying. 110. The moisture samples shall be open-piled in an oven and dried at a temperature of 100° C. until constant weight is attained, after which the oven-dry weight shall be determined.

Moisture Content. 111. The loss in weight expressed in percentage of the oven-dry weight as above determined shall be considered the moisture content of the specimen.

TOLERANCES

(*A*) *Weights.*

Weights. 112. The weight of test specimens and of moisture samples shall be determined to an accuracy of not less than 0.2 per cent.

(*B*) *Measurements.*

Measurements. 113. Measurements of test specimens will be made to the nearest 0.01 in. except in the case of tension parallel to grain, radial shrinkage, and tangential shrinkage, when measurements will be taken to the nearest 0.001 in.

(*C*) *Testing Machine Speeds.*

Testing Machine Speeds. 114. In no case shall the testing machine speed used vary more than 25 per cent from that specified for a given test. The load shall be applied continuously, without interruption, at the required speed throughout the test.

CALIBRATION

Calibration. 115. All apparatus used in obtaining data shall be calibrated at sufficiently frequent intervals to insure accuracy.[1]

[1] See the Standard Methods of Verification of Testing Machines (Serial Designation: E 4) of the American Society for Testing Materials, p. 921.

APPENDIX

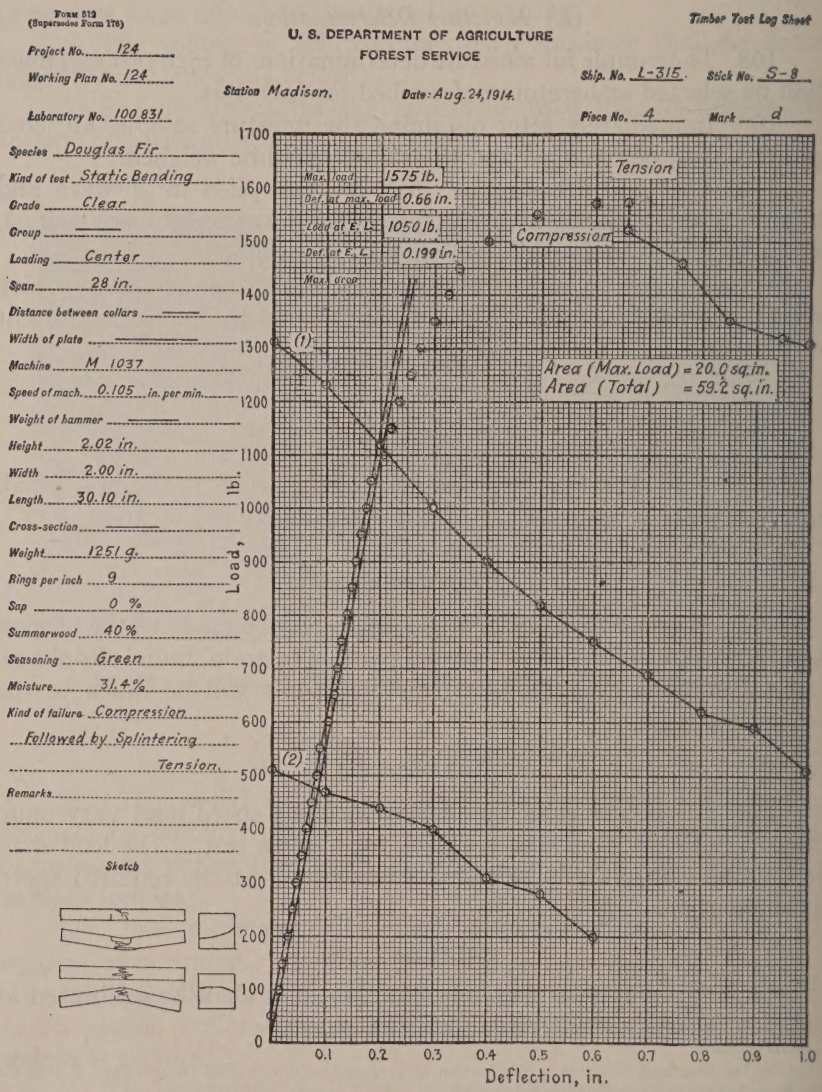


FIG. 1.—Sample Data Sheet for Static Bending Test.

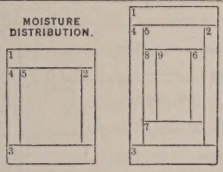
STATIC BENDING

L-315 (Ship No.) S-8 (Stick No.) Center Loading 100 831 (Lab. No.)
4 (Piece No.) d (Mark.) Station MADISON Date Aug. 24-'14 124 (Project No.)
Species Douglas Fir Grade Clear Seasoning Green
Rings 9 Sap 0 % Summerwood 40 % Moisture 31.4 %
Span 28 in. Length 30.10 in. Height 2.02 in. Width 2.00 in. Weight 1251 g.

SPECIFIC GRAVITY AS TEST.	SPECIFIC GRAVITY OV. DRY.	F. S. AT E. L.	M. OF R.	M. OF E.	SHEAR.	WORK TO E. L.	WORK TO MAX LOAD.	TOTAL WORK	
0.628	0.478	5410	8120	1756	292	0.92	7.1	20.9	

Rings: Up. ¼ Mid. ½ Low. ¼
Sum. wood: Up. ¼ Mid. ½ Low. ¼
Defects
Failure Compression Followed by Splintering Tension

MOISTURE DISTRIBUTION.



8—1434

FIG. 2.—Sample Computation Data Card for Static Bending Test.

COMPRESSION PARALLEL TO GRAIN

L-315 (Ship. No.) E-11 (Stick No.) 101 101 (Lab. No.)
1 (Piece No.) d-1 (Mark) Station Madison Date Aug. 25, 1914 124 (Project No.)
Species Douglas Fir Grade Clear Seasoning Green
Rings 9 Sap 90 % Summerwood 38 % Moisture 57.6 %
Length 8.00 in. Cross section 2.01 in. X 2.01 in. Weight 365 g.

SPECIFIC GRAVITY At Test	SPECIFIC GRAVITY Ov. Dry	MAX. LOAD	CRUSH. ST. AT E. L.	MAX. CRUSH. ST.	M. OF E.	LOAD AT E.L.	DEF. AT E.L
0.690	0.438	14 760	3220	3660	1992	13.000	0.0097

Defects
Failure Shear at Top

8—1463

FIG. 3.—Sample Computation Data Card for Compression-Parallel-to-Grain Test.

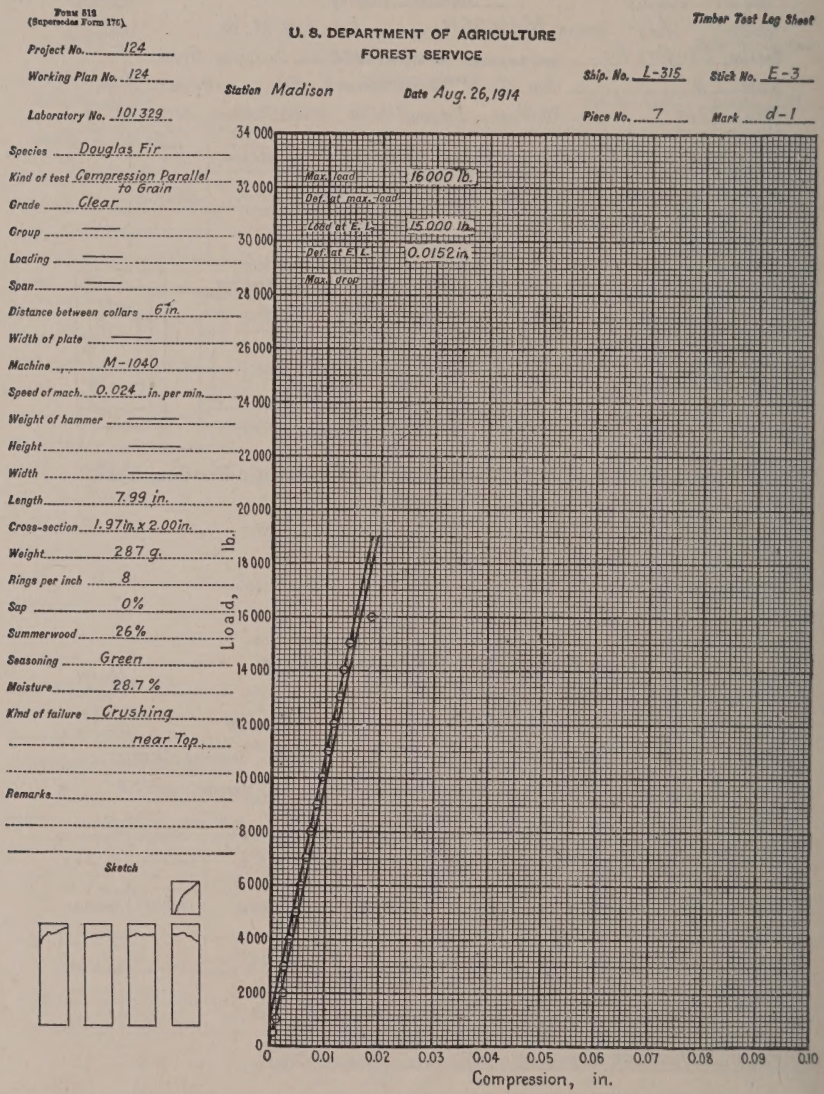


FIG. 4.—Sample Data Sheet for Compression-Parallel-to-Grain Test.

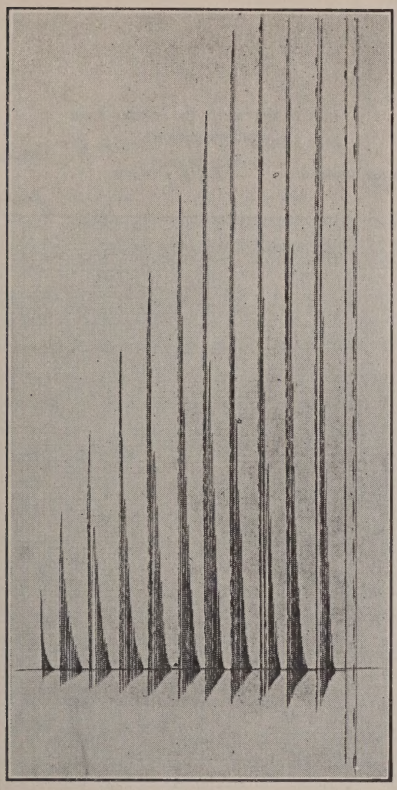


FIG. 5.—Sample Drum Record of Impact Test.

IMPACT BENDING

L-315 (Ship. No.) E-12 (Stick No.) 101151 (Lab. No.)
1 (Piece No.) C (Mark) Station MADISON Date Aug. 20, 1914 124 (Project No.)
Species Douglas Fir Grade Clear Seasoning Green
Rings 8 Sap 100 % Summerwood 30. % Moisture 61.4 %
Hammer 50 lbs. Span 28 in. Length 29.94 in. Height 2.00 in. Width 2.00 in. Weight 1370 g.

Drop No.	Head.	Def.	Def²	Set.	Drop No.	Head.	Def.	Def²	Set.		
1	1.0	0.13	0.017		11	12.0	0.50	0.250		Sp. Gr. (at test),	0.698
2	2.0	0.18	0.032		12	14.0	0.55	0.302		Sp. Gr. (oven dry),	0.432
3	3.0	0.22	0.048		13	16.0	0.62	0.384		F. S. at E. L.,	10 610
4	4.0	0.26	0.068		14	18.0	0.67	0.593		M. of E.,	1776
5	5.0	0.30	0.090		15					E. Resil.,	3.51
6	6.0	0.34	0.116		16					Max. Drop,	22 in.
7	7.0	0.36	0.130		17					a,	0.010
8	8.0	0.38	0.144		18					H	7.88
9	9.0	0.43	0.185		19					Δ	0.39
10	10.0	0.46	0.212		20						

Failure: Compression Followed by Splintering Tension.

S—1431

FIG. 6.—Sample Computation Data Card for Impact-Bending Test.

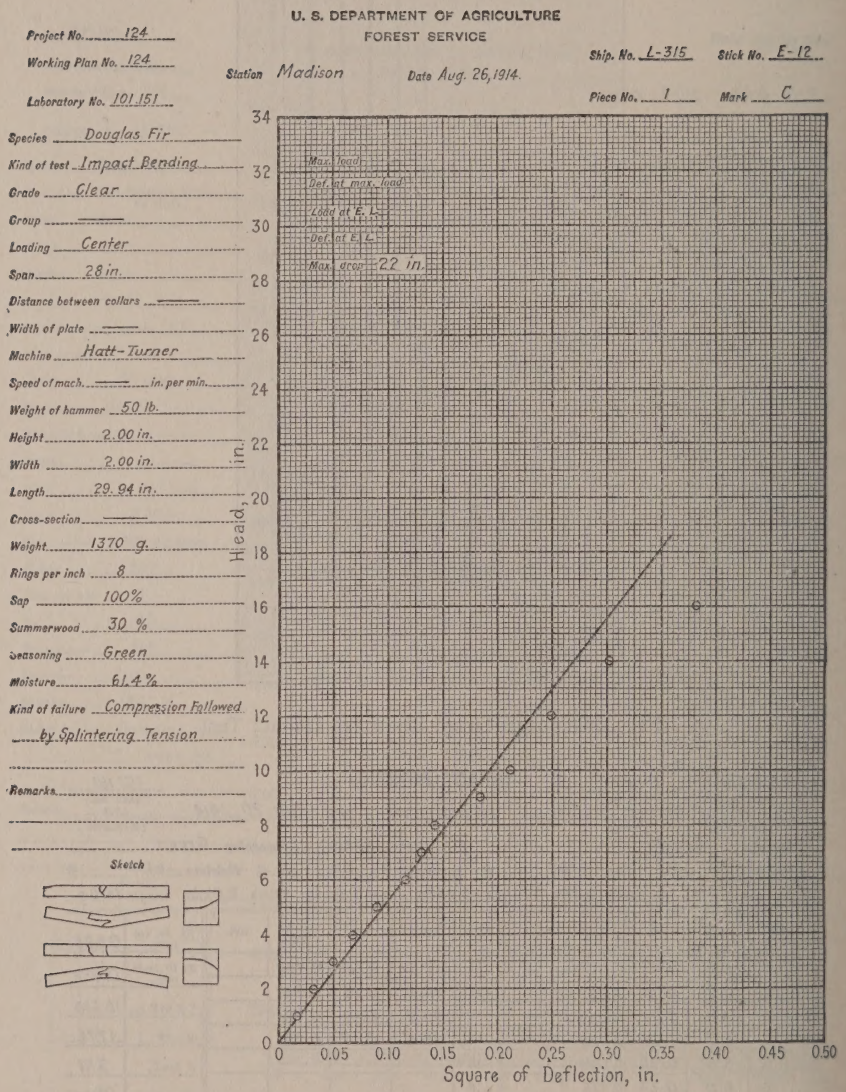


FIG. 7.—Sample Data Sheet for Impact-Bending Test.

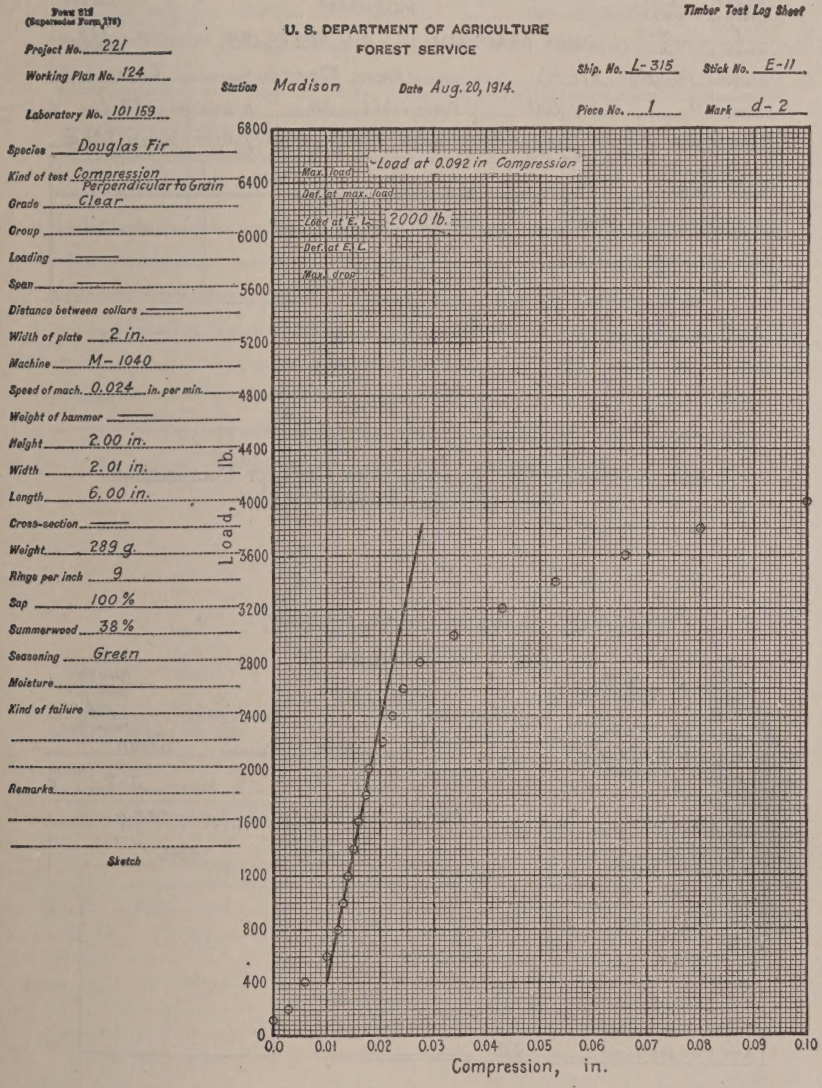


FIG. 8.—Sample Data Sheet for Compression-Perpendicular-to-Grain Test.

L-315 (Ship. No.) E-11 (Stick No.) COMPRESSION PERPENDICULAR TO GRAIN 101159 (Lab. No.)

1 (Piece No.) d-2 (Mark) Station MADISON Date Aug. 20, 1914 124 (Project No.)

Species Douglas Fir Grade Clear Seasoning Green

Rings 9 Sap 100 % Summerwood 38 % Moisture 62.1 %

Width of plate 2 in. Length 6.00 in. Height 2.00 in. Width 2.01 in. Weight 289 g.

Specific Gravity		Load at E. L.	Crush. St. at E. L.	$\Delta \div h_2$	
At Test.	Ov. Dry				
0.732	0.452	2000	498		

8—1464

FIG. 9.—Sample Computation Data Card for Compression-Perpendicular-to-Grain Test.

HARDNESS

L-315 (Ship. No.) E-10 (Stick No.) 101170 (Lab. No.)

1 (Piece No.) d-4 (Mark.) Station MADISON Date Aug. 20-14 124 (Project No.)

Species Douglas Fir Grade Clear Seasoning Green

Rings 8 Sap — % Summerwood 33 % Moisture 31.7 %

Length 6.01 in. Cross section 2.00 in. x 2.00 in. Weight 246 g.

	Specific Gravity.		Radial Surface.	Tangential Surface.	End Surface.
	At Test.	Ov. Dry.			
1	0.622	0.472	460	570	525
2			520	460	500
3					510
4					510
Avg.,			490	515	511
Avg. Rad. and Tang.			502		

Sketch.

8—1432

FIG. 10.—Sample Data and Computation Card for Hardness Test.

Tangential SHEAR

L-315 (Ship. No.) E-12 (Stick No.) 101183 (Lab. No.)

1 (Piece No.) C-5 (Mark) Station MADISON Date Aug. 21-14 124 (Project No.)

Species Douglas Fir Grade Clear Seasoning Green

Rings 8 Sap 75 % Summerwood 30 % Moisture 800 %

Shearing Area	Max. Load	Shearing Str.	Time		Sketch
2.02 x 2.01	3600	887			→

8—1465

FIG. 11.—Sample Data and Computation Card for Tangential-Shear-Parallel-to-Grain Test. (Surface of failure tangential.)

R TENSION PERPENDICULAR TO GRAIN

L-315 (Ship. No.) E-12 (Stick No.) 101189 (Lab. No.)

1 (Piece No.) C-6 (Mark) Station MADISON Date Aug. 21-14 124 (Project No.)

Species Douglas Fir Grade Clear Seasoning Green

Rings 8 Sap 80 % Summerwood 30 % Moisture 44.2 %

Height.	Width.	Length.	Maximum Load.	Tension. *Lbs. per sq. in.*
	2.01	0.97	533	273

Sketch.

8—2515

FIG. 12.—Sample Data and Computation Card for Radial-Tension-Perpendicular-to-Grain Test. (Surface of failure radial.)

R CLEAVAGE

1-315 (Ship. No.) E-12 (Stick No.) 101177 (Lab. No.)

1 (Piece No.) C-3 (Mark) Station MADISON Date Aug. 21, -14 124 (Project No.)

Species Douglas Fir Grade Clear Seasoning Green

Rings 8 Sap 75 % Summerwood 30 % Moisture 31.6 %

Height.	Width.	Length.	Max. Load.	Load per Inch Width.
	2.01	2.98	365	182

SKETCH.

8—1433

FIG. 13.—Sample Data and Computation Card for Radial-Cleavage Test. (Surface of failure radial.)

TENSION PARALLEL TO GRAIN

606 (Ship. No.) HM-14 (Stick No.) 347416 (Lab. No.)

2 (Piece No.) 5-F (Mark) Station MADISON Date Jan. 23, '19 221-1 (Project No.)

Species Douglas Fir Grade Clear Seasoning Green

Rings 10 Sap — % Summerwood 31 % Moisture 32.5 %

Cross Section.	Length.	Maximum Load.	Tension. Lbs. per sq. in.
0.641 x 0.617		4670	11810

8—2515

FIG. 14.—Sample Data and Computation Card for Tension-Parallel-to-Grain Test.

SPECIFIC GRAVITY AND VOLUMETRIC SHRINKAGE

L-315 (SHIP. NO.) S-6 (STICK NO.) 101197 (LAB. NO.)

1 (PIECE NO.) d-8 (MARK) STATION Madison, Aug. 20,-14 (DATE) 124 (PROJECT NO.)

SPECIES Douglas Fir

NOMINAL SIZE OF SPECIMEN 2 in. x 2 in. x 6 in. % SAP 0 % SUMMER WOOD 40

	DATE	RINGS PER INCH	WEIGHT, GRAMS	% MOIST	VOLUME C. C.	SPECIFIC GRAVITY	WEIGHT, LBS. PER CU. FT.	X % VOL. SHRINKAGE
GREEN	Aug. 20,-14	8	253	33.2	398	0.636	39.6	13.8
AIR DRY								
KILN DRY								
OVEN DRY	Sep. 25,-14		190		343	0.554	34.5	

X BASED ON ORIGINAL VOLUME (GREEN, AIR DRY, KILN DRY)

NOTE—USE BACK OF CARD FOR CARBON IMPRESSIONS

1ST WT. ----------

2D WT. ----------

REMARKS: ---------- VOL. ----------

8—3634

FIG. 15.—Sample Data and Computation Card for Specific Gravity and Shrinkage-in-Volume Test.

SHRINKAGE—RADIAL AND TANGENTIAL

L-315 (SHIP. NO.) (STICK NO) 101 200 101 199 (LAB. NOS.)

1 (PIECE NO.) d (MARK) STATION—MADISON, WISCONSIN 124 (PROJECT NO)

SPECIES Douglas Fir

NOMINAL SIZE OF SPECIMEN 1 in. X 4 in. x 1 in.

SEASONING	DATE	RINGS PER INCH	% SAP	% SUMMERWOOD	WIDTH INCHES	WEIGHT GRAMS	% MOISTURE	X % SHRINKAGE
RADIAL								
GREEN	Aug. 19-14	11	30	41	4.006	49.8	66.5	
OVEN-DRY	Oct. 5-14				3.808	29.9		4.9
TANGENTIAL								
GREEN	Aug. 19-14	12	95	34	4.016	64.0	119.1	
OVEN-DRY	Oct. 5-14				3.632	29.2		9.5

X BASED ON GREEN WIDTH

FIG. 16.—Sample Data and Computation Card for Radial and Tangential-Shrinkage Tests.

STANDARD METHODS

OF

CONDUCTING STATIC TESTS OF TIMBERS IN STRUCTURAL SIZES[1]

Serial Designation: D 198 - 27

These methods are issued under the fixed designation D 198; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1924; ADOPTED, 1927.

Although numerous tests of timber in structural sizes have been made for various purposes, there has been a general lack of uniformity of test procedure. Consequently, it has not been possible to correlate data from different sources, nor have the results obtained by different investigators contributed their full measure of usefulness toward an increasing fund of knowledge. Study and experience have shown the extent to which various factors, such as loading appliances, rate of loading, etc., influence test results, and it is with the thought of standardizing these and other factors that this test procedure is prepared. The plan has been to cover very definitely the tests of certain types of timbers such as bridge stringers and joists and at the same time to suggest procedure, so far as possible, for other structural timber forms. Summarized, the underlying purpose is the establishment of practice which will afford uniform results of the greatest ultimate value from all tests of timbers and at the same time permit the correlation of data so far as possible with existing data on timbers of certain species.

It is not the intention to discourage the use of methods other than those herein prescribed whenever the testing problem is of such nature as to warrant their application. However, experience has emphasized the importance of the requirements that have been specified in these methods, and variations in apparently minor details will often seriously affect the data obtained. It is recommended that any variations from the methods herein prescribed shall be recorded.

The field of timber testing is so broad, and involves so many objectives, particularly when built-up or manufactured articles are considered, that it is obviously difficult, if not impossible, to formulate correct procedure for all such cases. Ample opportunity, therefore, still awaits the application of initiative to special testing problems and promotion of progress in the art. It is hoped, however, that the principles incorporated in the proposed methods will at least serve to some extent as a guide in tests of even a special nature.

The test methods presented are the outgrowth of years of experience and research, largely on the part of the United States Forest Service. Such

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

sample data forms are included as were thought helpful to investigators in systematizing records. No attempt has been made to cover methods of computation or analysis, as these were considered to lie outside the scope of the standard. Data properly recorded can be analyzed at will, but fundamental differences in test procedure present a barrier beyond which analysis and correlation of results cannot proceed with any degree of assurance.

Purpose of Tests.

1. Tests of timbers in structural sizes are made to afford:

(*a*) Data upon which to base the formulation of grading rules and specifications.

(*b*) Data upon which to establish the relation between the strength properties of structural timbers and of small clear specimens cut from them, and for use in the establishment of allowable stresses.

(*c*) Data as to the influence of defects on the mechanical properties of timbers.

(*d*) Data on the strength properties of different species in structural sizes.

(*e*) Data for use in checking existing formulas relating to structural timbers.

(*f*) Data as to the influence of seasoning on the mechanical properties.

(*g*) Data as to the effect of form and shape of specimen on the properties.

(*h*) Data as to the influence of preservatives and methods of preservative treatment on the mechanical properties.

Classes of Tests.

2. In general, tests of timbers are divided into two classes, as follows:

(*a*) Major tests, that is, tests of principal members under investigation.

(*b*) Minor tests, that is, tests of small clear specimens cut from the principal members.

Kind of Tests.

3. (*a*) The principal tests of such structural timbers as bridge stringers or joists are: Static bending, compression parallel to grain, and compression perpendicular to grain.

(*b*) The accompanying minor tests on small clear specimens are: Static bending, compression parallel to grain, compression perpendicular to grain, hardness, and shear parallel to grain.

MATERIAL

COLLECTION

(*A*) *Selection*

Authentic Identification.

4. The material for test shall be selected by one qualified to identify the species.

Procedure for Bridge Stringers, Joists, etc.

5. In any given case, the number of test specimens shall be determined from due consideration of the purpose in mind, since definite instructions cannot be given to cover all cases. The following procedure applies particularly to full-size timbers, such as bridge stringers and joists, and incidentally to other types, as fabricated beams, to which special reference will be made in small-type paragraphs.

Number and Size of Major Specimens.

6. (*a*) For tests of timbers of any given species in structural sizes, such as bridge stringers, requiring a range in grade of material, at least forty 32-ft. or eighty 16-ft. green unsurfaced timbers should be selected in a manner that will provide representative material. The length of specimens required for test is dependent upon the depth. For suggested lengths of timbers other than bridge stringers, see Section 22.

(*b*) Timbers 8 by 16 in. or 6 by 12 in. in nominal cross-section are especially recommended.

Selection and Matching of Specimens.

7. Each 32-ft. timber shall be cut in half to furnish one 16-ft. specimen to be tested green and one to be tested air-dry, thus affording end matching. Where only 16-ft. material is available, the timbers shall be selected and matched in pairs, one timber to be tested green and the other to be tested air-dry. In matching these 16-ft. timbers all factors influencing their strength should, so far as practicable, be considered. Some of the most important factors are density, rate of growth, direction of grain (straight, diagonal, or spiral), character and location of defects (such as knots, checks, etc.), position of specimen in tree with respect to cross-section or height, and the characteristics of individual trees.

(*B*) *Field Marking*

Butt Designation.

8. At the time of selection a mark shall be made on each timber to designate the butt end.

Shipment Number.

9. All material collected at a given source and shipped at one time shall be given a shipment number or other designation, which shall be incorporated in the test records.

(*C*) *Field Descriptions*

Field Descriptions.

10. Field notes fully describing the material shall be carefully made by the collector. These notes shall, so far as possible, supply data as outlined in Table I and shall be forwarded for incorporation in the record.

(D) *Preparation for and Records of Shipment*

Preparation for Shipment.

11. (*a*) The ends of all green timbers shall be given a coating of paint or other suitable substance to retard end drying and end checking, care being taken to retain the butt designation. They shall be close-piled and before and during shipment shall be kept covered with damp shavings or other substance to prevent drying. If conditions are particularly favorable to the development of molds, stains, or decay, the specimens shall first be brushed or sprayed with a dilute solution of copper sulfate, or similar toxic solution.

(*b*) Record shall be made of the shipment routing, bill of lading, kind of shipment, date of shipment, and condition of material when shipped.

TABLE I.—SHIPMENT DESCRIPTION FIELD NOTES.

Project No., *XC* — Shipment No., *012*
Tree No., *1* — Species, *Longleaf pine*
Locality cut: State, *Florida* — County, *Nassau*
Township, *T3N: R25E* — Date cut, *April, 1914*
Slope, *Level.* Absolute elevation, *38 ft.* — Aspect, ...
Undergrowth, *Small amount—scattered* — Soil, *Sandy*
Height, *110 ft.* Seedling or sprout, *Seedling* — Age, *241 years*
Crown, *Large, well developed* — D.B.H., *26 in.*
Stem, *Clear, straight* — Date sawed, *May 12, 1914*
How and when transported from woods, *By wagon, May, 1914*

LIST OF SPECIMENS.

Piece No.	Size of Specimens			Volume, cu. ft.	Feetage, b. m.	Weight, lb. (Show date)
	Length, ft.	Width, in.	Depth, in.			
1	16.10	7.50	15.50	13.00	171	660

REMARKS: Note any other special points which would be of value.

(*c*) Record shall also be made of date of receipt of shipment at destination, its condition and the method of storage. The time elapsing between the sawing of green timbers and their testing should be reduced to a minimum.

DISPOSITION AT DESTINATION; MARKING AND STORAGE

Grouping.

12. When the material arrives at the testing laboratory, it shall be divided into two matched groups of timbers of approximately like quality as specified in Section 7, one portion to be tested green and the other to be weighed and placed under cover for air seasoning prior to testing. After the 32-ft. timbers have been cut into 16-ft. lengths, the specimens to be air-dried shall be selected alternately from butt and top cuts.

Marking.

13. The butt end of each 16-ft. specimen shall be designated "*A*" and the top end "*B*". Other symbols shall be given to each timber in classifying it as to density, position in tree, defects, or other factors that are under consideration. The minor test specimens and data forms shall carry such of these symbols as may be necessary to identify them fully.

Storage.

14. (*a*) The material to be tested green shall be close-piled on skids which are free from contact with the soil, and shall be kept covered. It shall not be held long enough to permit seasoning or damage from checking, decay, or insect attack.

(*b*) The ends of the specimens to be air-dried shall, if necessary, be given a suitable coat of pitch, paint, or other substance to retard checking. The specimens shall be open-piled in a place allowing free access and circulation of air, but shall be protected from sunshine, rain, snow, and moisture from the ground and shall not be subjected to artificial heat.

(*c*) All of the timbers to be air-dried shall be weighed when stored and at sufficiently frequent intervals thereafter to afford accurate data on the progress of seasoning. No material shall be considered thoroughly air-dry and fully conditioned for testing until practically constant weight has been reached.[1] (Wood absorbs and gives off moisture with changing atmospheric conditions; consequently it never comes to absolutely constant weight.)

(*d*) When properly seasoned, the timbers shall be surfaced on four sides before being tested.[2]

TESTS

KIND AND NUMBER

(*A*) *Major Tests*[3]

Major Bending.

15. (*a*) The structural member proper shall be tested as a beam. (See Sections 19 to 30.)

Compression Perpendicular to Grain.

(*b*) A section 60 in. long shall be cut from the "*A*" or butt end of each beam after test, except when end "*A*" fails by shear in the

[1] The many factors which influence the rate of seasoning, such as species, moisture content, and diverse climatic differences, preclude the establishment of a definite limit of loss in weight which may be permitted in green material prior to test.

[2] A desirable condition for testing air-dry material in small sizes may be considered as about 12-per-cent moisture content. Large timbers, such as bridge stringers, however, usually dry slowly, but may be considered conditioned when, during the season of most rapid drying, successive weighings indicate the moisture content is as low as may reasonably be expected, with due consideration to the climatic conditions of the locality. Such seasoning may require from about one to four years.

[3] The procedure outlined utilizes to best advantage the entire major specimen and if carefully followed will not usually admit the inclusion of specimens which have been seriously strained in the major bending test. The compression specimens referred to in Sections 15(*b*) and 15(*c*) necessarily include such defects as are normal to the material under test.

bending test; in which case the section shall be taken from the "*B*" or top end. A specimen 30 in. long for compression-perpendicular-to-grain test shall be cut from that end of the 60-in. piece which is nearer the point of loading.

(*c*) Two compression-parallel-to-grain specimens, each 6 by 6 by 24 in., shall be taken from the farther end of the 60-in. piece specified in Paragraph (*b*). **Compression Parallel to Grain.**

(*B*) *Minor Tests*[1]

16. (*a*) A 50-in. section shall be cut from the end of the beam opposite that required for the 60-in. specimen specified in Section 15 (*b*). Six 2 by 2 by 50-in. sticks shall be taken from this piece. In case defects make it difficult to obtain certain of the other clear specimens hereinafter provided for, they may be secured from the uninjured portion of the minor static bending specimens after the test covered in Paragraph (*b*) below. **Material.**

(*b*) Six static bending specimens free from defects shall be taken, one from each of the 50-in. sticks specified in Paragraph (*a*). **Static Bending.**

(*c*) Six compression-parallel-to-grain specimens free from defects shall be taken, one from each of the 50-in. sticks specified in Paragraph (*a*). **Compression Parallel to Grain.**

(*d*) Three compression-perpendicular-to-grain specimens free from defects shall be taken, one from each of three of the 50-in. sticks specified in Paragraph (*a*). **Compression Perpendicular to Grain.**

(*e*) Three hardness specimens free from defects shall be taken, one from each of three of the 50-in. sticks specified in Paragraph (*a*) other than those from which the specimens specified in Paragraph (*d*) are taken. **Hardness.**

(*f*) Twelve shear specimens free from defects shall be cut, two from each of the 50-in. sticks specified in Paragraph (*a*). One specimen from each pair taken from the same stick shall be tested in radial shear (surface of failure radial to growth rings) and the other in tangential shear (surface of failure tangential to growth rings). **Shear Parallel to Grain.**

GENERAL INSTRUCTIONS

(*A*) *Photographs of Timbers*

17. All air-dried timbers shall be photographed on four sides both before and after test. All green timbers shall be photographed **Timbers to be Photographed.**

[1] The procedure outlined utilizes to best advantage the entire major specimen and if carefully followed will not usually admit the inclusion of specimens which have been seriously strained in the major bending test. As specified in Section 26 of the Standard Methods of Testing Small Clear Specifications of the Timber (Serial Designation: D 143) of the American Society for Testing Materials, see p. 627, the minors selected shall be free from defects which will affect the properties under consideration.

on four sides after test only. The middle of the length of the beam, the load and support points, and the order of occurrence of the failures shall be indicated so as to show in the photograph; if necessary to bring them out in the photograph the failures should be penciled or pointed.

(*B*) *Moisture Control*

Control of Moisture Content.

18. (*a*) As specified in Section 14 (*c*), the major specimens to be tested in the air-dry condition shall be brought to practically constant weight before test. All tests shall be carried out in such a manner as to prevent large changes in moisture content.

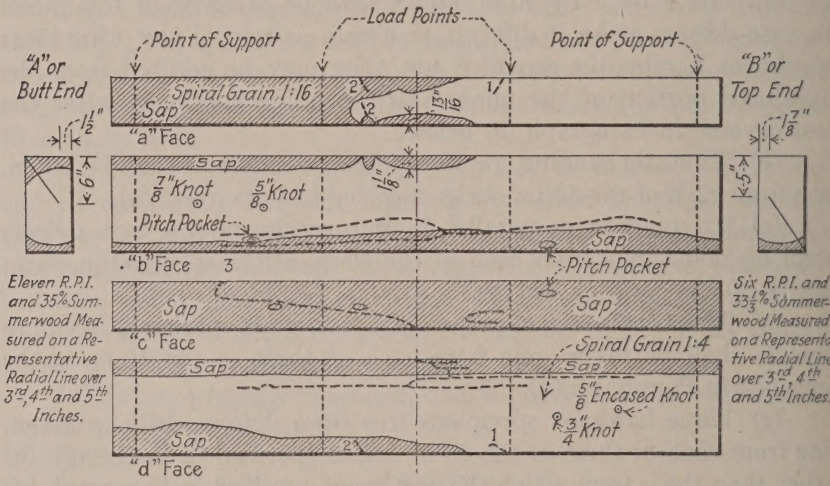


FIG. 1.—Sketch of Structural Size Timber Showing Method of Recording Location and Type of Defects and Other Pertinent Data.

Broken lines indicate test failures, and the accompanying numbers, their order of occurrence.

(*b*) All minor specimens shall be tested immediately after cutting from the majors to prevent change in moisture content.

PROCEDURE—MAJOR TESTS

(*A*) *Static Bending Test of Structural Size Timber*

Size and Weight.

19. (*a*) The beam shall be weighed before test. The length and the actual depth and width at the center shall be measured.

(*b*) For non-rectangular beams or beams non-uniform in section data shall be secured from which the exact size and shape of the section at any point throughout the length may be obtained.

Additional Marking.

20. In addition to the markings previously made, the four faces shall be lettered "*a*," "*b*," "*c*," and "*d*," beginning with *a* for the

face to which the load is applied and proceeding successively to the right (clockwise) when viewed from the "*A*" or butt end of the timber.

Sketch of Beam.

21. As part of the record, a sketch shall be made of the faces and ends of each beam showing the size, location, and type of defects, including cross-grain,[1] the distribution of sapwood and heartwood, the location of pith, when present, and the direction of the annual rings (see Fig. 1).[2] Detailed record shall also be made of other defects which may be present, such as crook, bow, cup, and twist.[3] The number of rings per inch and proportion of summerwood at both ends shall also be recorded.

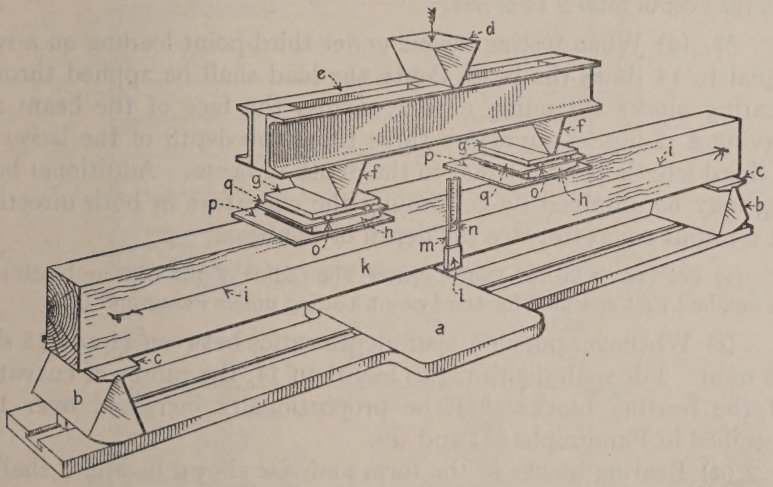


FIG. 2.—Diagrammatic Sketch Showing Method of Conducting Static Bending Test of Structural Sizes of Timber.

Loading, Span and Supports.

22. (*a*) For timbers in structural sizes such as bridge stringers and joists, third-point loading over a span length of 15 ft. shall be used. Knife edges rigidly attached to an auxiliary beam which in turn is hinged by a knife edge shall be used in applying the load. The supporting knife edges shall preferably be of the half-rocker type,

[1] Cross-grained wood is that in which the wood cells or fibers do not run parallel with the axis of the piece. Cross-grain may be classified as spiral, diagonal, dip, wavy, curly, and interlocking. Definitions of these terms may be found in U. S. Department of Agriculture *Circular No. 296*, p. 60. The presence of diagonal and spiral grain is usually indicated by surface checks; these invariably follow the direction of the grain. The amount of cross-grain in any portion of a timber may be expressed by the slope of the fibers (or checks) on each face with respect to the edges of the piece. Thus, a slope of 1 in 10 indicates a departure of the fibers from the direction of the axis of the specimen of 1 in. in 10 in. of length.

[2] Cross-section paper or quadrille-ruled note book paper is recommended for conveniently making this sketch of the beam to scale.

[3] For definitions of these terms, see U. S. Department of Agriculture *Circular No. 296*, p. 65.

and shall be so placed as to rock outward. To prevent accident or damage from their being thrust suddenly outward on failure of the specimen, they should be tied together by means of a slack chain or cable. Metal bearing plates 6 in. in width and not less than $\frac{1}{2}$ in. in thickness and extending entirely across the face of the beam shall be used over the supporting knife edges. (See Fig. 2.)

(*b*) When beams are tested with respect to particular uses, the dimensions shall be such as to simulate actual conditions as closely as practicable.

(*c*) Fabricated beams such as I or box types shall be full size or solid at the loading points without abrupt change from the solid to the normal beam section.

(*d*) Center loading shall not be used for beams over 4 in. in depth when the span-depth ratio is 14 or less.

Bearing Blocks.

23. (*a*) When testing beams under third-point loading on a span equal to 14 times the beam depth the load shall be applied through bearing blocks extending entirely across the face of the beam and having a radius of curvature three times the depth of the beam for a chord length at least equal to the depth of beam. Additional bearing may be obtained by continuing the curvature in both directions at a radius equal to twice the depth of the beam.

(*b*) For beams loaded at the center, the radius of the bearing block shall be one-half that specified for third-point loading under Paragraph (*a*).

(*c*) Whenever possible, span-depth ratios between 11 and 15 shall be used. For span-depth ratios less than 14, the radius of curvature of the bearing blocks shall be proportionally increased over that specified in Paragraphs (*a*) and (*b*).

(*d*) Bearing blocks of the form and size shown in Fig. 3 shall be used at the load points in testing timbers in structural sizes, such as 6 by 12-in. and 8 by 16-in. bridge stringers, on a 15-ft. span.

(*e*) Where the depth of beam is greater than 4 in., a thin steel plate slightly wider than the beam shall be placed between the loading block and the beam. For beams up to 6 in. in depth, the thickness of this plate shall be from $\frac{3}{32}$ to $\frac{1}{8}$ in. For greater depth the thickness of plates shall be increased slightly to about $\frac{5}{32}$ in. for timbers 6 by 12 in. or 8 by 16 in. in cross-section.

(*f*) For fabricated sections such as airplane I and box beams up to 6 in. in depth, the plate shall be from $\frac{1}{16}$ to $\frac{3}{32}$ in. in thickness.

(*g*) Metal bearing plates and rollers shall be used between each bearing block and its corresponding knife edge. The plates between rollers and knife edges shall be not less than $1\frac{1}{4}$ in. in thickness. If the supporting knife edges are of the full-rocker type rather than the

half-rocker type recommended in Section 22 (*a*), rollers shall be placed under one loading knife edge only.

Placement of Specimen in Machine.

24. The beam shall be placed in the testing machine symmetrically with respect to the supports. Parallelism shall be obtained at bearing points. In the case of warped beams, wedges or shims shall be used between the plate and the loading or supporting knife edges or both, as may be necessary to obtain an even bearing before the load is applied.[1] In the case of timbers containing defects, the load shall be applied to the poorer of the loading faces.

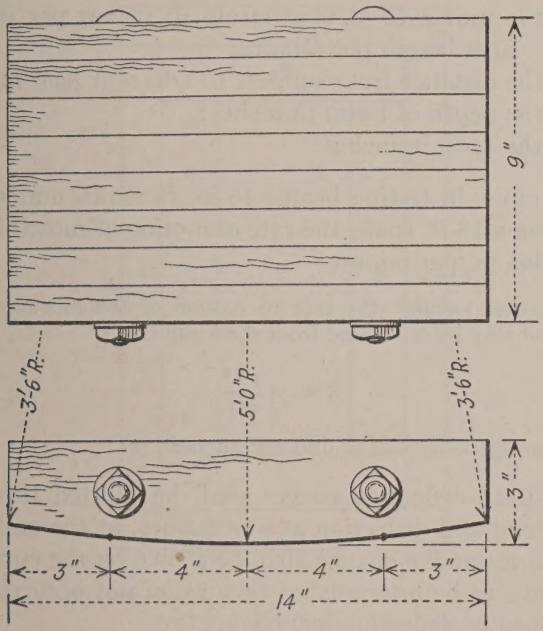


FIG. 3.—Details of Bearing Block for Timbers 12 to 16 in. in Depth When Tested in Third-Point Loading over 15-ft. Span.

Rate of Application of Load.

25. (*a*) The load shall be applied continuously, and with a uniform motion of the movable head throughout the test.

(*b*) *Case 1.*—Beams 4 in. or less in depth when containing defects, or 8 in. or less in depth when free from defects, shall be tested at a rate of fiber strain of 0.0015 in. per inch of outer fiber length per minute.[2]

[1] The use of shims or wedges is recommended in preference to planing because in certain cases the latter procedure may effect changes in section where undesirable.

[2] For tolerance on rate of motion of movable head see Section 51.

Case 2.—Beams over 4 in. in depth when containing defects, or over 8 in. in depth when free from defects shall be tested at a rate of fiber strain of 0.0007 in. per inch of outer fiber length per minute.[1]

(*c*) For two symmetrical concentrated loads the rate of motion of the movable head of the testing machine may be calculated from the formula:

$$N = \frac{z\,a}{3\,d}(3\,L - 4\,a)$$

where N = the rate of motion of moving head in inches per minute;
z = the unit rate of fiber strain in inches per inch of outer fiber length per minute;
a = the distance from support to adjacent load in inches;
d = the depth of beam in inches;
L = the span in inches.

For example, in testing beams 16 in. in depth under third-point loading and on a 15-ft. span, the rate of motion of movable head shall be 0.26 ±0.065 in. per minute.[1]

(*d*) For center loading, the rate of motion of the movable head of the testing machine may be calculated from the formula:

$$N = \times \frac{z\,L^2}{6\,d}$$

where the same legend applies as under Paragraph (*c*).

Load-Deflection Curves.

26. (*a*) Load-deflection curves shall be plotted throughout the test.[2] The load and deflection at first failure, at the maximum load, and at points of sudden change shall be shown on the curve, although they may have to be interpolated because of not occurring at one of the regular load or deflection increments.

(*b*) The test shall be continued until the maximum load has been well passed. In the event of horizontal shear, the maximum load may occur after such failure.

Method of Determining Deflection.

27. (*a*) Deflections of the beam at mid-height at the center of the span with respect to points at mid-height immediately above the supports shall be taken to the nearest 0.01 in.

(*b*) Deflection may be read directly by means of a wire stretched between nails driven at mid-height of the beam immediately above the supports and extending across a scale attached at mid-height at the center of the span. Fig. 2 illustrates the method of test. A

[1] Tolerance in accordance with Section 51.

[2] See Fig. 1 of the Appendix for a sample static-bending data sheet and Fig. 2 of the Appendix for a sample computation data card.

telescope or reading glass will be found useful in observing the deflections. When a reading glass is used parallax shall be prevented by placing a reflecting surface adjacent to the scale in order that the wire and its image may be made to coincide.

(*c*) Where special refinement is desired, deflection readings may be taken simultaneously on both sides of the beam, the wires (Paragraph (*b*)) being supported over free running pulleys and held taut with small suspended weights.

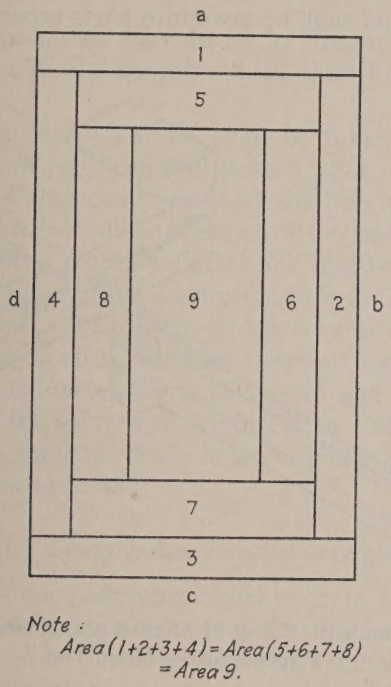


FIG. 4.—Cross-Section of Timber Showing Method of Cutting Up Moisture Distribution Sections.

Description of Failures.

28. The failures shall be described in detail as to type, manner and order of occurrence and position in beam. The descriptions of the failures shall be suitably recorded and correlated with the load-deflection curves. The failures shall be sketched on the drawing of the beam referred to in Section 21, with notations as to the order of their occurrence. The section of the beam containing the failure shall be held for examination and reference until analysis of the data has been completed.

Identification Sample.

29. A section of the beam at least 2 in. in length shall be retained for purposes of identification and future reference. The number of rings per inch and proportion of summerwood shall be recorded for this section.

Specimens for Moisture Determinations.

30. Two sections 1 in. in length shall be cut from near the place of failure for moisture determinations. One of these shall be used for determining the average moisture content of the test specimen. The other, which may be termed a moisture-distribution section, shall be used for determining the moisture content of different portions of the cross-section and shall be sawn into parts according to the pattern

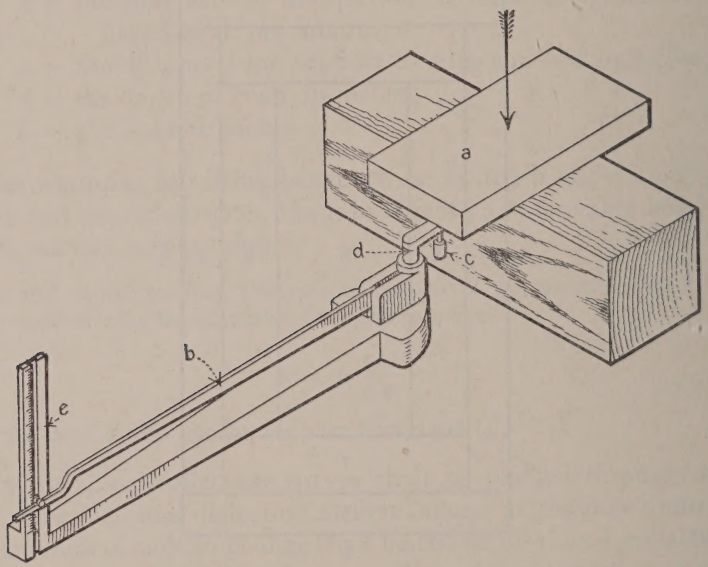


FIG. 5.—Diagrammatic Sketch of Method of Conducting Compression-Perpendicular-to-Grain Test.

shown in Fig. 4. Moisture determinations in each case shall be made in accordance with the procedure specified in Sections 44 to 48.

(*B*) *Compression Perpendicular to Grain*

Size of Specimen.

31. The compression-perpendicular-to-grain tests shall be made on specimens 30 in. in length and of the cross-section of the major beam. The actual width and length shall be measured and recorded, and all defects shall be described.

Loading.

32. The load shall be applied through a metal plate 6 in. in width placed across a clear face of the specimen at the center of the length and at right angles to it. The load shall be applied to a face

which corresponds to either the loading or supporting face of the static bending specimen. (Section 24.) (See Fig. 5.)

33. (*a*) The load shall be applied continuously throughout the test at a rate of motion of movable head[1] determined from the formula:[2] **Rate of Application of Load.**

$$N = 0.0175\, d^{\frac{4}{9}}$$

where N = the rate of motion of moving head in inches per minute;
and
d = the depth of beam in inches.

(*b*) For the specimens 8 by 16 in. in cross-section, for example, the rate of descent of the movable head shall be 0.060 ± 0.015 in. per minute.[3]

34. Load-compression curves[4] shall be taken for all specimens until the elastic limit has been well passed, after which the test shall be discontinued. Compression readings shall be taken to 0.001 in. **Load-Compression Curves.**

35. The specimen shall be weighed after test and two sections shall be cut near the place of failure for moisture determinations. One of these shall be used for determining the average moisture content of the specimen. The other, which may be termed a moisture distribution section, shall be used for determining the moisture content of different portions of the cross-section and shall be sawn into parts according to the pattern shown in Fig. 4. Moisture determinations in each case shall be made in accordance with the procedure specified in Sections 44 to 48. **Weight and Moisture Content.**

(*C*) *Compression Parallel to Grain*

36. The major compression-parallel-to-grain tests shall be made on the 6 by 6 by 24-in. specimens prepared from the pieces specified in Section 15 (*c*). The actual cross-section dimensions, the rate of growth, and the percentage of summerwood shall be determined and recorded for each specimen. All defects shall be described. **Size of Specimen.**

37. Care shall be exercised in preparing the compression-parallel-to-grain test specimens to make the end surfaces plane, parallel, and normal to the axis of the specimen.[5] **End Surfaces.**

---

[1] For tolerance on rate of motion of movable head see Section 51.

[2] Tests are now in progress to establish more definitely the proper rate of application of load; therefore, the requirement of this section may be changed on the completion of the study.

[3] Tolerance in accordance with Section 51.

[4] See Fig. 4 of the Appendix for a sample compression-perpendicular-to-grain data sheet and Fig. 3 of the Appendix for a sample computation data card.

[5] A sharp fine-toothed saw of either the cross-cut or "novelty" cross-cut type is indispensable for obtaining smooth end surfaces. Power equipment with accurate table guides is especially recommended for this work, though miter-box hand sawing is permissible. Hand planing of the ends, if carefully done is sometimes advisable.

Spherical Bearing.

38. (*a*) A spherical bearing block shall be used to prevent eccentricity of loading. The radius of the sphere shall be as small as practicable, in order to facilitate adjustment of the bearing plate to the specimen. The suspended type of spherical bearing, to be used on top of the specimen, is especially recommended. The size of the compression plate shall be slightly larger than is necessary to encompass the end of the specimen when the latter is centered on the head.

(*b*) The adjustment of the suspended spherical bearing block may be accomplished by keeping the compression plate in motion about its bearing as contact is made with the specimen.

(*c*) In the spherical bearing block for use on the weighing table, adjustment shall be made by moving the bearing plate with the specimen centered thereon.

Rate of Application of Load.

39. The load shall be applied continuously throughout the test at a rate of motion of the movable head of 0.036 ± 0.009 in. per minute.[1]

Records.

40. The maximum load obtained shall be recorded, together with a description and sketch of the failure. The section of the specimen containing the failure shall be held for examination and reference until analysis of the data has been completed.

Position of Test Failures.

41. In order to obtain satisfactory and uniform results, the failures shall be made to develop in the body of the specimen. With specimens of uniform cross-section this may be obtained by having the ends properly prepared and dried to a slightly lower moisture content than the body. With green material, it will usually suffice to close-pile the specimens, cover the body with a damp or wet cloth, and expose the ends for a short time. For air-dry material it may sometimes be advisable, should the failures in test indicate that a slight end drying is necessary, to pile the specimens in a similar manner and place them in a desiccator.

Weight and Moisture Content.

42. The specimen shall be weighed after test, and a moisture section shall be cut from the body of the piece near the failure. Moisture determinations shall be made in accordance with the procedure specified in Sections 44 to 48.

PROCEDURE—MINOR TESTS

Minor Test Procedures.

43. The procedure for the conducting of minor tests shall conform to the procedure specified in the Standard Methods of Testing Small Clear Specimens of Timber (Serial Designation: D 143) of the American Society for Testing Materials.[2]

[1] Tolerance in accordance with Section 51.

[2] See p. 627.

MOISTURE DETERMINATIONS

Selection of Samples.

44. The samples for moisture determinations shall be selected as hereinbefore described for each test.

Cutting of "Distribution" Sample.

45. The moisture-distribution specimens shall be cut into sections according to Fig. 4, as specified in Sections 30 and 35.

Weighing of Moisture Sample.

46. Immediately upon obtaining any moisture sample, all loose splinters shall be removed and the sample weighed.

Drying.

47. The moisture samples shall be open-piled in a ventilated oven and dried at a temperature which shall ultimately be increased to 98° C. ± 3° C. until constant weight is attained, after which the resulting weight shall be carefully determined and recorded as the oven-dry weight. For specimens not over 1 in. in length (in the direction of grain) about 48 hours' continuous drying under the conditions specified is sufficient to bring the specimens to constant weight. For drying longer specimens, considerably more time will be required, since the rate of drying from the side grain surface of wood is only about one-sixth that from the end surface.

Moisture Content.

48. The loss in weight expressed as a percentage of the oven-dry weight as above determined shall be considered the moisture content of the specimen.

TOLERANCES

(*A*) *Weights*

Weights.

49. The weight of test specimens and of moisture samples shall be determined with an error of not more than 0.2 per cent.

(*B*) *Measurements*

Measurements.

50. Measurements of test specimens shall be made to the nearest 0.01 in.

(*C*) *Testing Machine Speeds*

Testing Machine Speeds.

51. In no case shall the testing machine speed used vary more than 25 per cent[1] from that specified for a given test. The load shall be applied continuously at the required speed throughout the test.

CALIBRATION

Calibration.

52. All apparatus used in obtaining data shall be calibrated at sufficiently frequent intervals to insure accuracy.[2]

---

[1] Tests by the U. S. Forest Service show that this speed limitation is necessary in order that the stress shall not vary more than 1 per cent due to variation in rate of fiber strain.

[2] See the Standard Methods of Verification of Testing Machines (Serial Designation: E 4) of the American Society for Testing Materials, p. 921.

APPENDIX

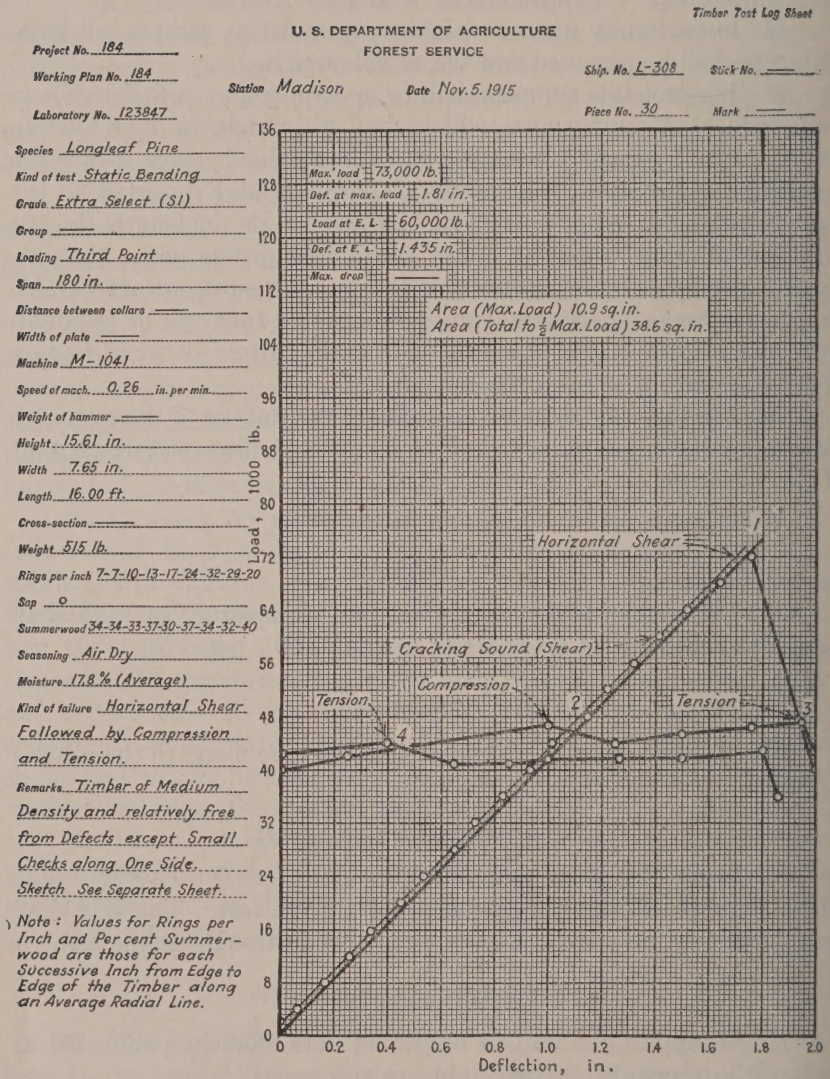


FIG. 1.—Sample Data Sheet for Static Bending Test.

Form 507.
(Revised January, 1918.)

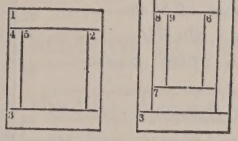
STATIC BENDING

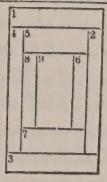
L-308 (Ship No.) — (Stick No.) One third points. Loading 12X847 (Lab. No.)
30 (Piece No.) — (Mark.) Station MADISON, WIS. Date 11-5-15 184 (Project No.)
Species Longleaf Pine Grade No 1 Seasoning Air dry
Rings 7-7-10-13-17-24-32-29-20 Sap 0 % Summerwood 34-34-33 % Moisture 17.9 %
37-30-37-34-32-40 %
Span 180 in. Length 16.00 ft. Height 15.61 in. Width 7.85 in. Weight 515½ #

SPECIFIC GRAVITY. AS TEST.	SPECIFIC GRAVITY. OV. DRY.	F. S. AT E. L.	M. OF R.	M. OF E.	SHEAR.	WORK TO E. L.	WORK TO MAX. LOAD.	TOTAL WORK.	
.622	.528	5820	7080	1781	458	1.74	3.2	11.5	

Rings: Up. ¼ — Mid. ½ — Low. ¼ —
Sum. wood: Up. ¼ — Mid. ½ — Low. ¼ —
Defects —
Failure Horizontal Shear followed by Compression and tension.

MOISTURE DISTRIBUTION.





8—1454
19998M

FIG. 2.—Sample Computation Data Card for Static Bending Test.

(Revised January, 1915)

COMPRESSION AT RIGHT ANGLES TO GRAIN

308 (Ship No.) — (Stick No.) 124509 (Lab. No.)
30 (Piece No.) — (Mark) Station MADISON, WIS. Date 11-30-15 184 (Project No.)
Species Longleaf Pine Grade 1 Seasoning Air dry
Rings 20 Sap 0 % Summerwood 35 % Moisture 17.4 %
Width of plate .522 Length 30.11 in. Height 15.56 in. Width 7.51 in. Weight 76 #

SPECIFIC GRAVITY At Test.	SPECIFIC GRAVITY Ov. Dry	LOAD AT E. L.	CRUSH. ST. AT E. L.	Δ ÷ h	
.598	.510	38,000 #	854	—	

8—1464
19908M

FIG. 3.—Sample Computation Data Card for Compression-Perpendicular-to-Grain Test.

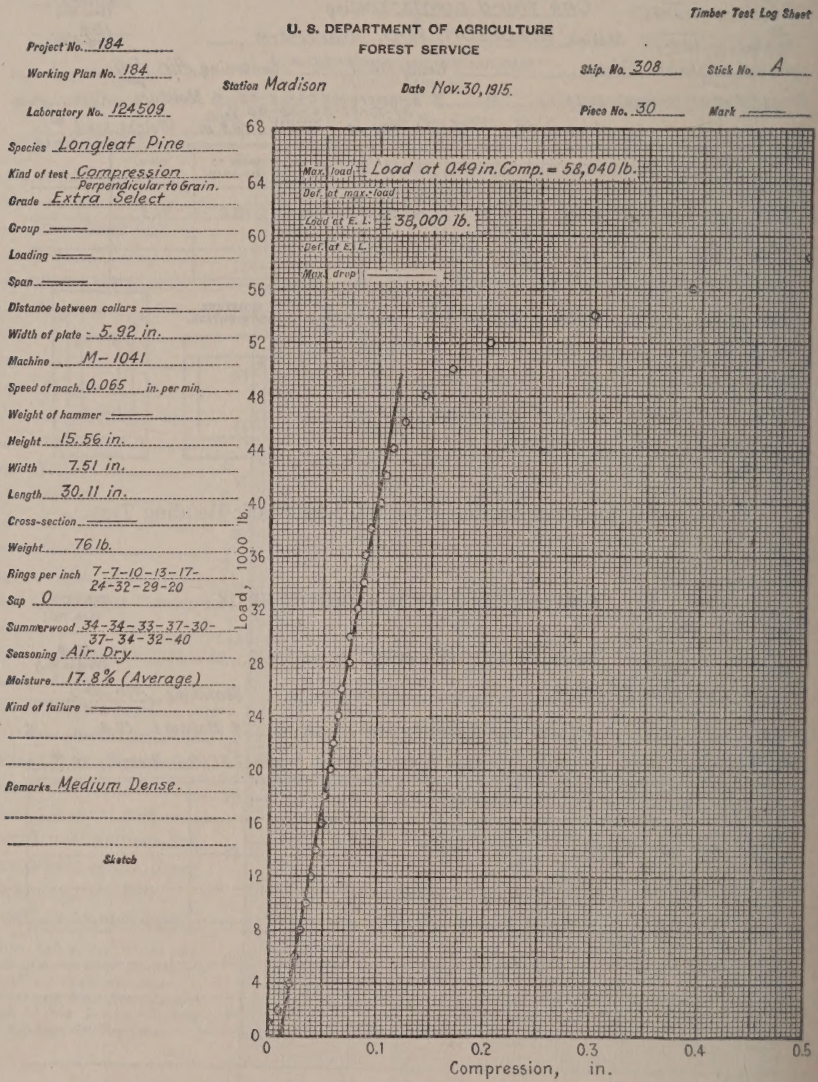


FIG. 4.—Sample Data Sheet for Compression-Perpendicular-to-Grain Tests.

STANDARD METHODS
OF
SAMPLING AND ANALYSIS OF CREOSOTE OIL[1]

Serial Designation: D 38 - 27

These methods are issued under the fixed designation D 38; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1915; ADOPTED, 1917; REVISED, 1918, 1924, 1927.

SAMPLING

Continuous-Drip Sample.

1. Wherever the oil is being loaded or discharged by means of a pump the following method shall be used:

A ½-in. sampling pipe shall be inserted in the line through which the oil is being pumped, on the discharge side of the pump, preferably in a rising section of the pipe line. This sampling pipe shall extend one-half way to the center of the main pipe and with the inner open end of the sampling pipe turned at an angle of 90 deg. and facing the flow of the liquid. This pipe shall be provided with a plug cock and shall discharge into a receiver of 50 to 100 gal. capacity. The plug cock shall be so adjusted that, with a steady continuous flow of the oil, the receiver shall be filled in the time required to pump the entire shipment. The receiver shall be provided with a steam coil sufficient to keep the contents at a temperature not exceeding 120° F. Immediately upon completion of the pumping, the contents of the receiver shall be very thoroughly agitated and a duplicate 1-qt. sample taken immediately for the test. The amount of the drip sample collected shall be not less than one gallon for each 1000 gal. of oil handled, except in the case of large boat shipments, where a maximum of 100 gal. is sufficient.

ANALYSIS

WATER

Apparatus.

2. (*a*) *Still.*—A vertical, cylindrical copper still, with removable flanged top and yoke, of the form and approximate dimensions shown in Fig. 1, shall be used.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

(*b*) *Thermometer.*—The thermometer shall conform to the following requirements. These specifications cover a total immersion thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being 0 to 400° C. or 30 to 760° F., respectively.

TYPE: Etched stem, glass.
LIQUID: Mercury.
RANGE AND SUBDIVISION: 0 to 400° C. in 1° C. or 30 to 760° F. in 2° F.
TOTAL LENGTH: 378 to 384 mm. (14.88 to 15.12 in.).
STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).
BULB: Corning normal or equally suitable thermometric glass.
Length, 10 to 15 mm. (0.39 to 0.59 in.).
Diameter, 5.0 to 6.0 mm. (0.20 to 0.24 in.).
DISTANCE TO 0° C. OR 32° F. LINE FROM BOTTOM OF BULB: 25 to 35 mm. (0.98 to 1.38 in.).
DISTANCE TO 400° C. OR 752° F. LINE FROM TOP OF THERMOMETER: 30 to 45 mm. (1.18 to 1.77 in.).
FILLING ABOVE MERCURY: Nitrogen gas.
TOP FINISH: Glass ring.
GRADUATION: All lines, figures, and letters clear cut and distinct. The first and each succeeding 5° C. or 10° F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.
IMMERSION: Total.
SPECIAL MARKING: "A.S.T.M. High Distillation," a serial number and the manufacturer's name or trade mark shall be etched on the stem.
SCALE ERROR: The error at any point of the scale up to 370° C. or 700° F. when the thermometer is standardized as provided below shall not exceed 1° C. or 2° F.
STANDARDIZATION: The thermometer shall be standardized immersed in the testing bath to the top of the mercury column, at the ice point and at temperature intervals of approximately 50° C. or 100° F. up to 370° C. or 700° F.
TEST FOR PERMANENCY OF RANGE: After being subjected to a temperature between 360 and 370° C. or 680 and 700° F. for 24 hours, the accuracy shall be within the limit specified.
CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. High Distillation, 0 to 400° C." or "A.S.T.M. High Distillation, 30 to 760° F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

NOTE.—Inasmuch as those dimensions given above which differ from the corresponding dimensions that appeared in Methods D 38 until their revision in 1924 are of such a nature as to affect the facility of making but not the accuracy of the thermometer, any thermometer now in use complying with the former require-

ments of D 38 shall be accepted as satisfactory by producers and users of creosote oil; provided that all thermometers hereafter purchased shall be in accordance with the new requirements.

(*c*) *Condenser.*—A copper trough condenser shall be used, with straight-walled glass tube, having approximately the form and dimensions shown in Fig. 2.

(*d*) *Separatory Funnel.*—A separatory funnel of the form shown in Fig. 2 shall be used, having a total capacity of 120 cc., and the outlet graduated in fifths of a cubic centimeter.

3. The apparatus shall be set up as shown in Fig. 2. **Assembling.**

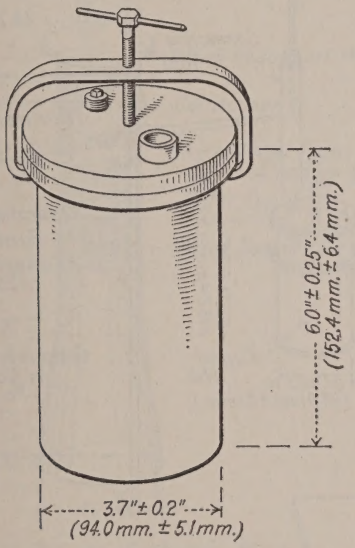


FIG. 1.—Copper Still

4. When any measurable amount of water is present in the distillate below 210° C. as determined in accordance with the Tentative Method of Test for Distillation of Cresote Oil (Serial Designation: D 246 – 27 T) of the American Society for Testing Materials,[1] the oil and water in this fraction shall be separated, if possible, and measured separately. If more than 2 per cent of water is present, or if the water is apparently present to an extent in excess of 2 per cent, but an accurate separation is impossible, the percentage of water present shall be determined by the following method, and the water-free oil so obtained shall be used in the distillation test: **Water Test.**

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 531.

Measure 200 cc. of oil in graduated cylinder, and pour into copper still, allowing the cylinder to drain into the still for several minutes. Attach lid and clamp, using a paper gasket slightly wet with oil around the flange of the still. Apply heat by means of the ring burner, which shall be placed just above the level of the oil in the still at the beginning of the test, and gradually lowered when most of the water has distilled over. Continue the distillation until the

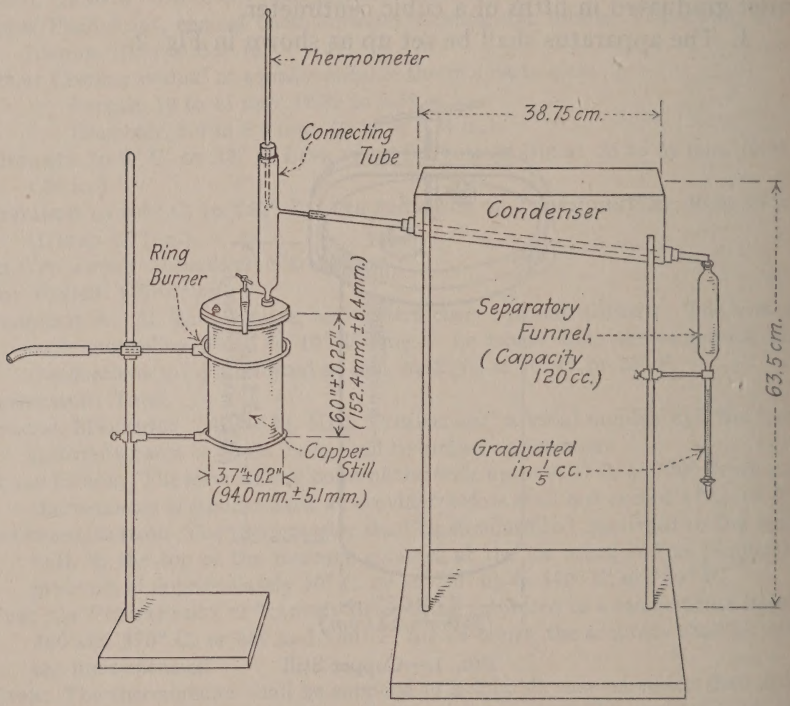


FIG. 2.—Assembled Apparatus for Water Test.

vapor temperature, indicated by the thermometer with the bulb opposite the offtake of the connecting tube, reaches 205° C. Collect distillate in separatory funnel. When the distillation is completed, and a clear separation of water and oil in the funnel has taken place, read the water by volume and draw off; and return any light oil distilled over with the water to the oil in the still. The dehydrated oil from the still shall be used for the distillation test.

Alternative Test for Water.

5. The amount of water may be determined in accordance with the Standard Method of Test for Water in Petroleum Products and

Other Bituminous Materials (Serial Designation: D 95) of the American Society for Testing Materials).[1]

NOTE.—This method is of particular service where the determination of water only is involved and the dehydrated material is not to be used subsequently for analysis.

MATTER INSOLUBLE IN BENZOL

6. (*a*) *Extractor.*—The extractor may be of the form shown in Fig. 3, or any similar form in which the oil is subjected to direct washing by the boiling vapors of the solvent. Apparatus.

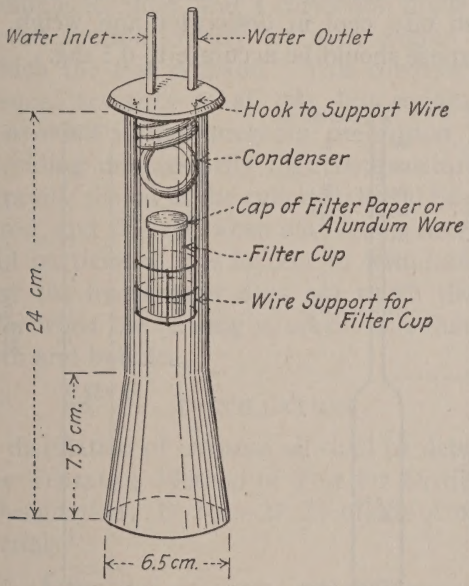


FIG. 3.—Extraction Flask.

(*b*) *Filtering Medium.*—The filtering medium may be either two thicknesses of S. & S. No. 575 hardened filter paper, 15 cm. in diameter, arranged in cup shape by folding symmetrically; or alundum thimbles, flat bottom, 30 x 80 RA 98. If filter papers are used, prior to using they shall be soaked in benzol to remove any grease, dried in a steam oven and kept in a desiccator until ready to be used. The filter-paper cup may be suspended in the extractor flask by a wire basket hung from two small hooks on the under surface of the metal cover of the flask. If the alundum thimble is used it may be supported by making two perforations in the top of the thimble, and suspending from the cover by German silver or platinum wires.

[1] See p. 433.

"Insoluble in Benzol" Test.

7. Weigh 10 g. of dry oil in a 100-cc. beaker. Add about 50 cc. of pure benzol and transfer at once to the filter cup. The filter cup or thimble is previously weighed, and the paper cup shall always be kept in a weighing bottle until ready for use. Wash out the beaker with benzol, passing all washings through the filter cup, and place latter at once in the extraction apparatus. Extractor shall contain a suitable quantity of pure benzol. Sufficient heat to boil the solvent shall be provided by means of an electric heater or a steam bath. Continue the extraction until the descending solvent is practically colorless, and remove the filter cup and dry in steam oven until all solvent is driven off; cool in desiccator and weigh. The balance used for this purpose should be accurate to 0.5 mg.

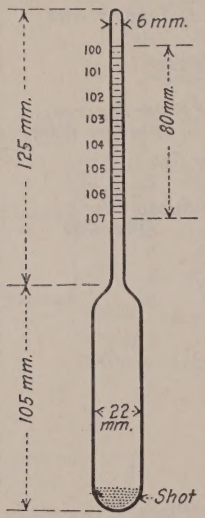


FIG. 4.—Hydrometer.

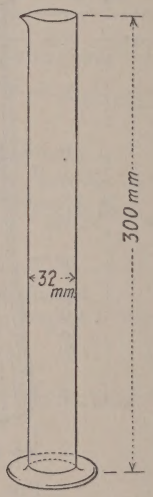


FIG. 5.—Cylinder.

SPECIFIC GRAVITY

Apparatus.

8. (*a*) *Hydrometer.*—The hydrometer shall be of the form shown in Fig. 4. It shall have the following dimensions:

Length of stem	125 mm.;	permissible variation,		12.5 mm.
" " bulb	105 "	"	"	10.5 "
" " scale	80 "	"	"	8.0 "
Diameter of stem	6 "	"	"	0.5 "
" " bulb	22 "	"	"	2.0 "

A set of two hydrometers with ranges 1.00 to 1.08 and 1.07 to 1.15 will suffice.

(*b*) *Cylinder.*—The cylinder shall be of the form shown in Fig. 5. It shall have the following dimensions:

Length	300 mm.;	permissible variation,	30 mm.
Diameter	32 "	"	"	3 "

Specific Gravity Test.

9. The oil shall be brought to a temperature of 38° C. (100° F.), and the determination shall be made at that temperature unless the oil is not entirely liquid at 38° C. In case the oil requires to be brought to a higher temperature than 38° C. in order to render it completely fluid, it shall be tested at the lowest temperature at which it is completely fluid, and a correction made by adding 0.0008 to the observed specific gravity for each degree Centigrade above 38° C. at which the test is made. This correction factor does not apply with equal accuracy to all oils, but serious error due to its use will be avoided if the foregoing precaution is observed, with respect to avoiding unnecessarily high temperature. Before taking the specific gravity the oil in the cylinder should be stirred thoroughly with a glass rod, and this rod when withdrawn from the liquid should show no solid particles at the instant of withdrawal. Care should be taken that the hydrometer does not touch the sides or bottom of the cylinder when the reading is taken, and that the oil surface is free from froth and bubbles.

DISTILLATION

Distillation.

10. The distillation of creosote oil shall be determined in accordance with the Tentative Method of Test for Distillation of Creosote Oil (Serial Designation: D 246 – 27 T) of the American Society for Testing Materials.[1]

SPECIFIC GRAVITY OF FRACTIONS

Specific Gravity of Fractions.

11. As specific gravity is an absolute physical determination, any recognized method which can be applied to the quantity and quality of material at hand to be tested must be considered satisfactory. The following methods are convenient and accurate means for the relatively small amounts of oil available in determining specific gravity of fractions to be tested.

(*a*) *Liquid Fractions.*

Apparatus.

12. The Westphal balance may be used.

Specific Gravity of Liquid Fractions.

13. If the fraction to be tested is liquid at a temperature not exceeding 60° C., the Westphal balance can be used with convenience and rapidity. A special type of Westphal balance is obtainable,

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 531.

designed for testing very small quantities. However, the ordinary type of Westphal balance can be adapted to testing small fractions by the use of a special plummet. When using the ordinary balance with the special plummet, extra care is needed that the adjustment of the balance be accurately made. The plummet can readily be made in the laboratory from a piece of ordinary glass tubing 7 mm. in outside diameter, sealed at the end, and melting into the glass where sealed a short platinum wire. After cooling, place 9 to 10 g. of mercury in the tube, making a column 35 to 40 mm. high. Seal off the tube within 20 mm. of the top of the mercury column with blowpipe flame. The plummet shall have a length of about 55 to 60 mm. over all, and shall weigh between 10 and 12 g.

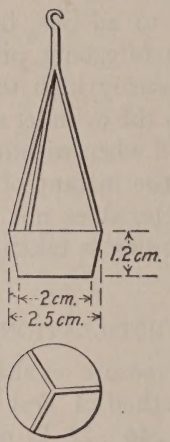


FIG. 6.—Platinum or Nickel Pan.

(*b*) *Solid and Semi-solid Fractions.*

Apparatus.

14. A pan of the form shown in Fig. 6, having the following approximate dimensions, may be used:

Diameter of base	20 mm.
Diameter of top	25 "
Depth	12 "
Diameter of wire	1 "
Total weight	7 g.

The pan and wires are made of platinum or nickel.

Specific Gravity of Solid and Semi-Solid Fractions.

15. Solid or semi-solid fractions of oil which cannot be readily liquefied can be rapidly and accurately tested in this apparatus by the usual method of weighing in air and in water. The usual precautions of igniting the pan before use, and avoiding the enclosure of air or water in the sample, should be observed.

NOTE.—The method for liquid fractions is usually applicable to the fractions 235 to 315° C. and the method for solid and semi-solid fractions to the fractions 315 to 355° C.

FLOAT TEST OF RESIDUE

Float Test of Residue.

16. The residue remaining in the retort after the distillation test shall remain until it reaches a temperature between 100 and 125° C. The consistency of the residue shall then be determined in accordance with the Standard Method of Float Test for Bituminous Materials (Serial Designation: D 139) of the American Society for Testing Materials.[1]

NOTE.—Care must be taken at the end of the distillation test to see that the vapor temperature as indicated on the thermometer does not rise above 355° C.; an excess temperature of only 1 or 2° C. at this point makes the float test invalid.

COKE RESIDUE

Coke Residue.

17. The coke residue of creosote oil shall be determined in accordance with the Standard Method of Test for Coke Residue of Creosote Oil (Serial Designation: D 168) of the American Society for Testing Materials.[2]

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[1] See p. 506. [2] See p. 692.

STANDARD METHOD OF TEST
FOR
COKE RESIDUE OF CREOSOTE OIL[1]

Serial Designation: D 168 - 27

This method is issued under the fixed designation D 168; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED, 1927.

Apparatus. 1. *Crucible.*—The crucible shall be of platinum and shall have a capacity of 20 to 30 cc. The cover of the crucible shall be of the inverted type, having a depth of about 1 cm., the wall tightly fitting the crucible except for a slight crease.

Procedure. 2. The residue resulting from the distillation test, carried out in accordance with the Tentative Method of Test for Distillation of Creosote Oil (Serial Designation: D 246 - 27 T) of the American Society for Testing Materials,[2] shall be poured directly into the tared crucible or into a tin box wherein it may be heated on a water or steam bath, but not over a flame. About one gram of the residue shall be weighed into the covered crucible and then placed on a platinum, nichrome, or fire-clay triangle over a Bunsen burner, with the bottom of the crucible 6 to 8 cm. from the top of the burner. The burner flame shall be regulated to a height of 20 cm. while burning free and the crucible shall be exposed to the full flame for 7 minutes. A Meker burner may be used, or the crucible may be heated for several minutes in an electric furnace. Whatever the method of heating employed, the temperature during the entire 7-minute period shall be not less than 950° C. and should be as near that temperature as possible. At the end of this period the flame shall be removed, the crucible transferred to a desiccator and permitted to cool after which it shall be weighed. The residue in the crucible after ignition shall be reported as "fixed carbon" (that is, coke).

Precautions. 3. The test shall be conducted in a part of the laboratory free from draughts.

Calculations. 4. The percentage of coke obtained in accordance with Section 2 shall be calculated on the basis of the original sample of oil.

Example.—With a retort distillation of 29 per cent of residue at 355° C., the residue containing 28 per cent of fixed carbon:

$$\text{Coke in oil} = \frac{29 \times 28}{100} = 8.1 \text{ per cent}$$

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 531.

STANDARD METHODS OF CHEMICAL ANALYSIS OF ZINC CHLORIDE[1]

Serial Designation: D 199 - 27

These methods are issued under the fixed designation D 199; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1924; ADOPTED, 1927.

These methods cover the determination of the percentages of insoluble basic zinc chloride, zinc chloride, and iron present in commercial concentrated solutions of zinc chloride or in fused or granulated zinc chloride, for use in the preservative treatment of wood.

PREPARATION AND STANDARDIZATION OF SOLUTIONS

Standard Ferrocyanide Solution.—Weigh out 43.25 g. of c.p. potassium ferrocyanide and 14 g. of c.p. crystallized sodium sulfite, dissolve in water and make up to 1 liter at room temperature. Shake thoroughly. Standardize against a zinc solution of known concentration prepared from spelter of known zinc content or from c.p. zinc oxide which has been previously ignited. One cubic centimeter of this solution will be equal to approximately 0.01 g. of zinc. The standardization should be carried out as nearly as possible in the same manner as in the estimation of zinc and approximately the same amount of zinc should be present. Keep the solution in a dark bottle. Shake thoroughly before each using and standardize each time it is used.

Uranium Acetate Indicator.—Dissolve 4.4 g. of c.p. uranium acetate, free from sodium, in 100 cc. of hot water and 2 cc. of glacial acetic acid. Use this as an external indicator on a paraffined plate making the drops as nearly 0.05 cc. as possible.

Hydrogen Peroxide.—The usual laboratory reagent is satisfactory, if fresh.

Hydrogen Sulfide Solution.—The usual laboratory reagent is satisfactory.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

DETERMINATION OF INSOLUBLE OR BASIC ZINC CHLORIDE

Weigh from 10 to 14 g. of the sample, if fused or granulated, from a stoppered weighing bottle, or place an equivalent quantity, if a solution, into a 600-cc. beaker. Add cold water to 400 cc. Stir the contents of the beaker until solution is complete. Allow to settle over night. Filter the solution through a 12.5-cm. filter paper which has previously been washed, dried, and weighed. Receive filtrate in a 1000-cc. graduated measuring flask. Policeman the beaker, and wash the insoluble matter in the filter paper until the filtrate measures 1 liter. Dry the filter paper containing the insoluble matter over night in an oven heated to 100° C. (212° F.). Cool and weigh between clipped watch glasses. Calculate the increase in weight of filter paper to a percentage of the original sample.[1]

Duplicate determinations should check within 1.3 per cent.

DETERMINATION OF ZINC

(*a*) *Volumetric method.* (For use when manganese chloride does not exceed 0.3 per cent.)

The filtrate obtained from the estimation of insoluble basic zinc chloride and whose volume is exactly 1 liter is shaken and three aliquot portions of 100 cc. each are taken with an accurate 100-cc. pipette and transferred into 450-cc. Griffins beakers. Add to each portion 15 g. of ammonium chloride and 5 cc. of concentrated hydrochloric acid. Dilute to 350 cc. and heat nearly to boiling. Titrate slowly with vigorous stirring using a solution of potassium ferrocyanide as the standard reagent and uranium acetate as an external indicator. The average of the three aliquot portions should be reported.

(*b*) *Volumetric Method.* (For use when the manganese chloride equals or exceeds 0.3 per cent.)

To the aliquot portions taken as described under (*a*), 1 cc. of hydrogen peroxide (2 to 3 per cent) and 10 cc. of ammonia (1:1) shall be added. Stand on steam bath until settled. Filter off the manganese, wash beaker and paper twice with hot water. Dissolve the precipitate in the smallest amount of hydrochloric acid (1:1) in the original beaker, heat until all is dissolved; the volume of the solution should be about 20 cc. Reprecipitate the manganese with 1 cc. of hydrogen peroxide and 10 cc. of ammonia, boil, filter, and wash several times with hot water. Add the filtrate to that obtained in the first separation. Add 15 cc. of concentrated hydrochloric acid

[1] A Gooch crucible may be used in place of the weighed filter paper.

to the combined filtrate and just neutralize with concentrated ammonia, then add 5 cc. of hydrochloric acid in excess. Dilute to 325 cc. and add 25 cc. of saturated hydrogen sulfide water to remove any traces of hydrogen peroxide, heat and titrate as in (*a*).

(*c*) *Gravimetric Method.* (Alternate method, for use when only a limited amount of work is necessary.)

The filtrate obtained from the estimation of insoluble basic zinc chloride and whose volume is exactly 1 liter is shaken and three aliquot portions of 200 cc. each are taken with an accurate pipette and transferred to 450-cc. Griffins beakers. Add to each 4 cc. of concentrated c.p. sulfuric acid. Evaporate on a steam bath; then on a steam plate or hot plate to copious SO_3 fumes, to completely eliminate chlorides. Cool and take up in 100 cc. of hot distilled water. Add 0.5 g. of aluminum powder. Cover with a watch glass. Heat to boiling and boil 5 minutes. Filter through an 11-cm. filter paper. Receive the filtrate in a covered 1000-cc. Griffins beaker. Wash the beaker and filter thoroughly with hot water until a drop of methyl orange indicator placed behind the double fold of filter paper shows no acidity. Exactly neutralize the filtrate with dilute ammonia. Use great care and precision, and carry the neutralization just to the end point.

Add 10 cc. of 0.1 *N* sulfuric acid (3 cc. of concentrated c.p. sulfuric acid in 1000 cc. of water). Dilute to 650 cc. Cover the beaker, and bubble a rapid stream of hydrogen sulfide for one-half to one hour at room temperature.

Settle and filter through a double filter of one 15-cm. and one 11-cm. paper[1] folded together. Transfer the precipitate to the filter paper. Thoroughly policeman the beaker until the zinc sulfide is all removed except a very thin film which clings tenaciously to the glass at the surface of the liquid. Thoroughly wash the beaker and precipitate at room temperature with water saturated with hydrogen sulfide. Repeat washing of filter paper, and precipitate five or six times. Transfer paper and precipitate to an ignited, cooled, desiccated and weighed porcelain crucible of suitable capacity (about 25 to 30 cc.). Carefully dry the paper and precipitate and when dry completely burn off the paper at as low a temperature as possible. When carbon has been completely burned out, ignite the resultant oxide of zinc strongly to as high a temperature as is available, but not higher than can be attained with a laboratory blast lamp with the aid of gas and air. Heat for 30 minutes. After strong ignition, cool the crucible, desiccate and weigh. The increase in weight is zinc oxide. The

[1] Ashless paper should be used.

weight of the zinc oxide multiplied by the factor 1.6749, multiplied by 100, divided by the weight of sample in the aliquot portion taken, equals the percentages of zinc chloride.

The average of the results obtained with the three aliquot portions analyzed shall be reported. Results should agree within 1.5 per cent.

ESTIMATION OF IRON AND ALUMINA

Weigh 10 g. of the sample, if fused or granulated, or place an equivalent quantity, if a solution, into a suitable beaker and dissolve in 100 cc. of water or dilute to 100 cc., if a solution. Add sufficient hydrochloric acid to dissolve any basic zinc chloride. Add a slight excess of bromine water and boil off excess. Neutralize with a weak solution of sodium carbonate until a permanent precipitate of zinc carbonate is obtained. Add three drops of glacial acetic acid and 2 g. of sodium acetate, and boil. Filter and wash. Redissolve the precipitate in the original beaker with hot hydrochloric acid (1: 1). Reprecipitate the iron and alumina with a slight excess of ammonia, filter and wash free from chlorine. Ignite in a platinum crucible and weigh as $Fe_2O_3+Al_2O_3$.

STANDARD DEFINITIONS
OF
TERMS RELATING TO STRUCTURAL TIMBER

Serial Designation: D 9 – 15

These definitions are issued under the fixed designation D 9; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1907; REVISED, 1915.

DEFINITION OF STRUCTURAL TIMBER

By the term "Structural Timber" is understood all such products of wood in which the strength of the timber is the controlling element in their selection and use. The following is a list of products which are recommended for consideration as structural timbers:

Trestle Timbers.—Stringers, caps, posts, mud sills, bracing, bridge ties, guard rails.

Car Timbers.—Car framing, including upper framing; car sills.

Framing for Buildings.—Posts, mud sills, girders, framing joists.

Ship Timbers.—Ship timbers, ship decking.

Cross Arms for Poles.

STANDARD DEFECTS

Measurements which refer to the diameter of knots or holes should be considered as referring to the mean or average diameter.

1. *Sound Knot.*—A sound knot is one which is solid across its face and which is as hard as the wood surrounding it; it may be either red or black, and is so fixed by growth or position that it will retain its place in the piece.

2. *Loose Knot.*—A loose knot is one not firmly held in place by growth or position.

3. *Pith Knot.*—A pith knot is a sound knot with a pith hole not more than $\frac{1}{4}$ in. in diameter in the center.

4. *Encased Knot.*—An encased knot is one whose growth rings are not intergrown and homogeneous with the growth

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

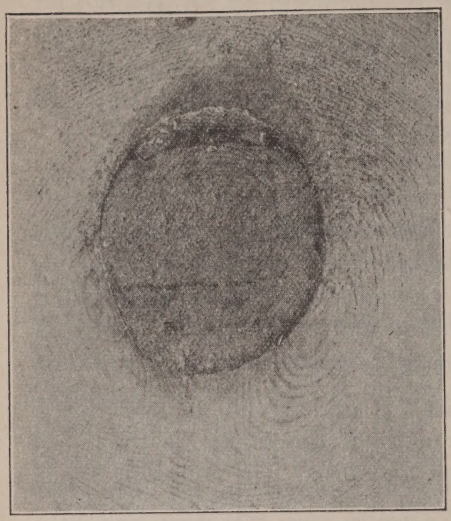

FIG. 1.—Loose Knot.

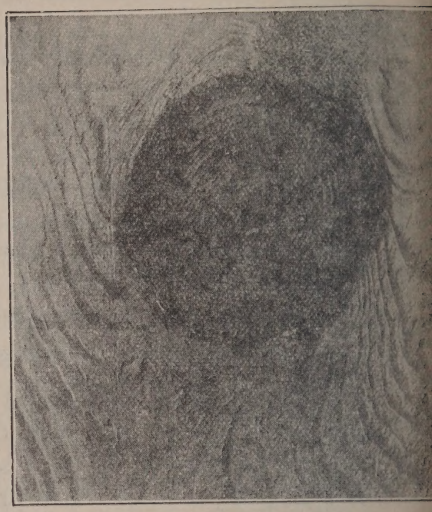

FIG. 3.—Encased Knot.

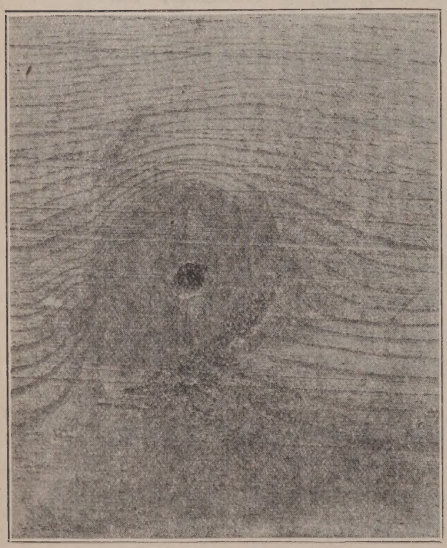

FIG. 2.—Pith Knot.

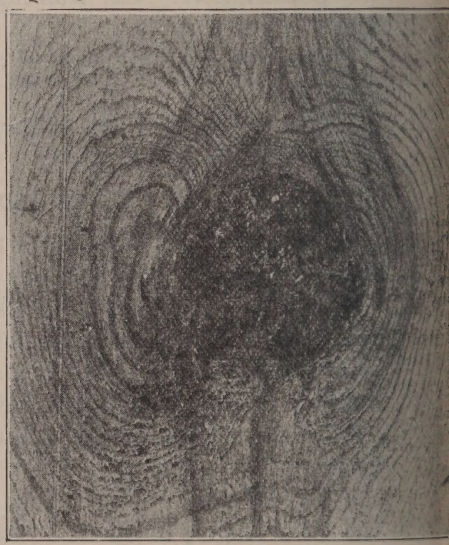

FIG. 4.—Rotten Knot.

rings of the piece it is in. The encasement may be partial or complete; if intergrown partially or so fixed by growth or position that it will retain its place in the piece, it shall be considered a sound knot; if completely intergrown on one face, it is a watertight knot.

5. *Rotten Knot.*—A rotten knot is one not as hard as the wood it is in.

6. *Pin Knot.*—A pin knot is a sound knot not over $\frac{1}{2}$ in. in diameter.

7. *Standard Knot.*—A standard knot is a sound knot not over $1\frac{1}{2}$ in. in diameter.

8. *Large Knot.*—A large knot is a sound knot, more than $1\frac{1}{2}$ in. in diameter.

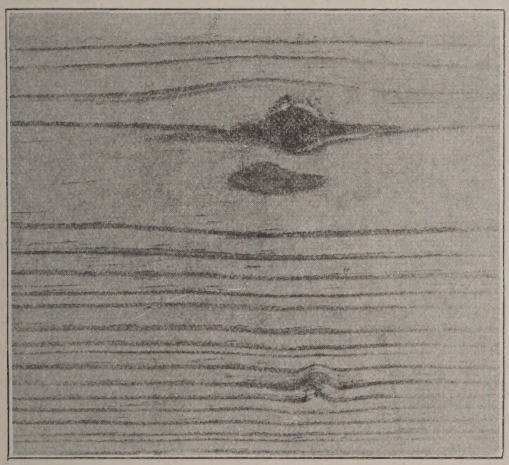

FIG. 5.—Pin Knot.

9. *Round Knot.*—A round knot is one which is oval or circular in form.

10. *Spike Knot.*—A spike knot is one sawn in a lengthwise direction; the mean or average width shall be considered in measuring these knots.

11. *Pitch Pockets.*—Pitch pockets are openings between the grain of the wood containing more or less pitch or bark. These shal be classified as *small*, *standard* and *large* pitch pockets.

(*a*) *Small Pitch Pocket.*—A small pitch pocket is one not over $\frac{1}{8}$ in. wide.

(*b*) *Standard Pitch Pocket.*—A standard pitch pocket is one not over $\frac{3}{8}$ in. wide, or 3 in. in length.

(*c*) *Large Pitch Pocket.*—A large pitch pocket is one over $\frac{3}{8}$ in. wide, or over 3 in. in length.

12. *Pitch Streak.*—A pitch streak is a well-defined accumulation of pitch at one point in the piece. When not sufficient to develop a well-defined streak, or where the fiber between grains, that is, the coarse-grained fiber, usually termed "spring wood," is not saturated with pitch, it shall not be considered a defect.

13. *Wane.*—Wane is bark, or the lack of wood from any cause, on edges of timbers.

14. *Shakes.*—Shakes are splits or checks in timbers which usually cause a separation of the wood between annual rings.

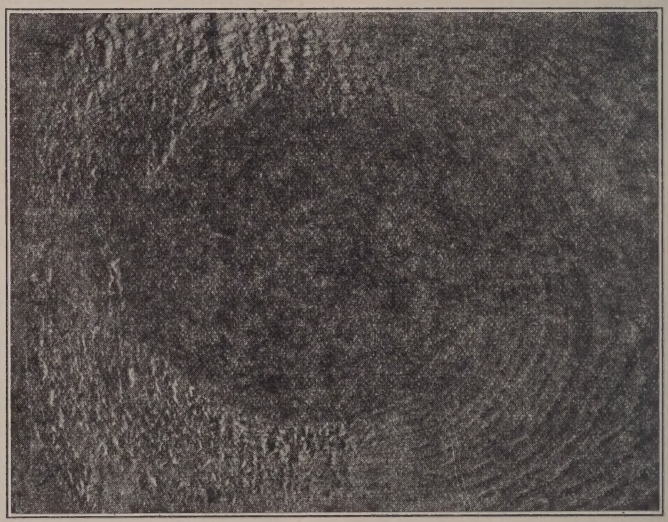

FIG. 6.—Standard Knot.

15. *Rot, Dote and Red Heart.*—Any form of decay which may be evident either as a dark red discoloration not found in the sound wood, or the presence of white or red rotten spots, shall be considered as a defect.

16. *Ring Shake.*—An opening between the annual rings.

17. *Through Shake.*—A shake which extends between two faces of a timber.

STANDARD NAMES FOR STRUCTURAL TIMBERS

1. *Southern Yellow Pine.*—This term includes the species of yellow pine growing in the southern states from Virginia to

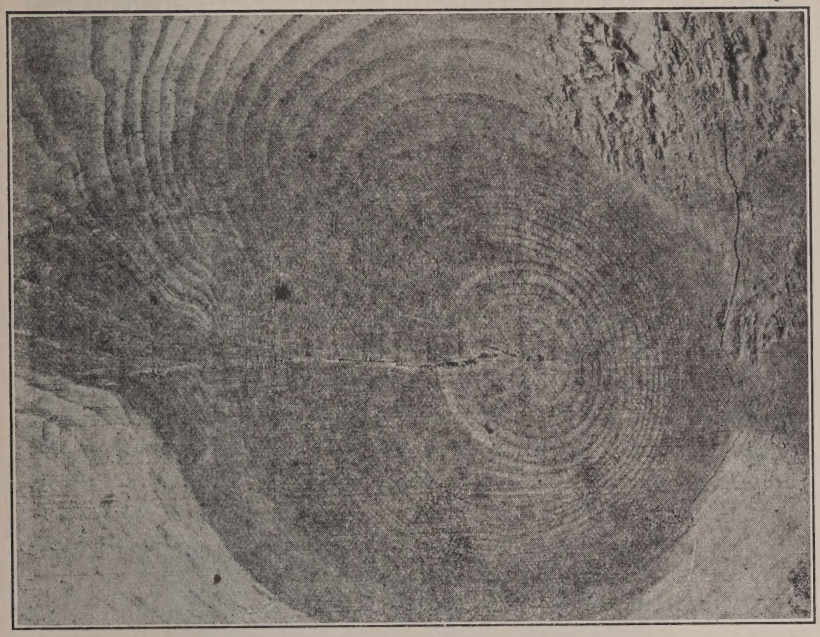

FIG. 7. Large Knot.

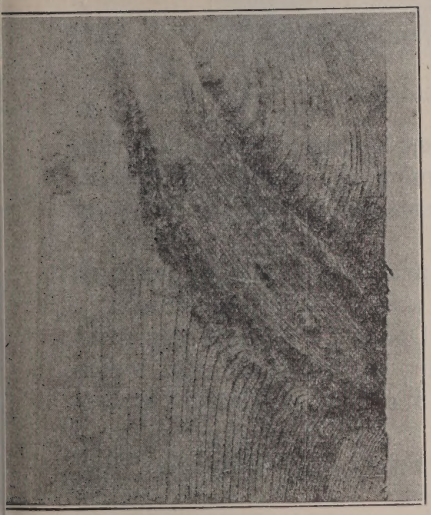

FIG. 8.—Spike Knot.

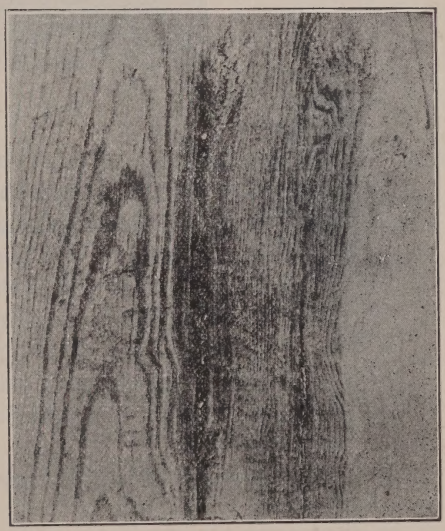

FIG. 9.—Pitch Pocket.

Texas, that is, the pines hitherto known as longleaf pine (*Pinus palustris*), shortleaf pine (*Pinus echinata*), loblolly pine (*Pinus taeda*), Cuban pine (*Pinus heterophylla*) and pond pine (*Pinus serotina*).

Under this heading, two classes of timber are designated: (*a*) dense southern yellow pine and (*b*) sound southern yellow pine. It is understood that these two terms are descriptive of quality rather than of botanical species.

(*a*) Dense southern yellow pine shall show on one end or the other an average of at least six annual rings per inch and at least one-third summer wood, or else the greater number of the rings shall show at least one-third summer wood, all as measured over the third, fourth, and fifth inches on a radial line from the pith. Wide-ringed material excluded by this rule will be acceptable, provided that the amount of summer wood as above measured shall be at least one-half.

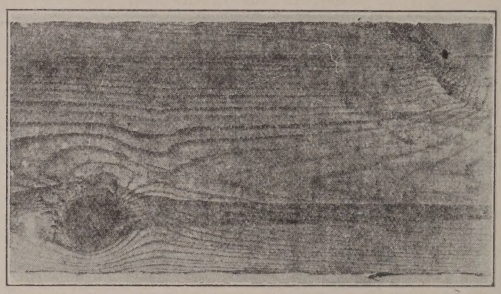

FIG. 10.—Pitch Streak.

The contrast in color between summer wood and spring wood shall be sharp and the summer wood shall be dark in color, except in pieces having considerably above the minimum requirement for summer wood.

(*b*) Sound southern yellow pine shall include pieces of southern pine without any ring or summer-wood requirement.

2. *Douglas Fir.*—The term "Douglas Fir" is to cover the timber known likewise as yellow fir, red fir, western fir, Washington fir, Oregon or Puget Sound fir or pine, northwest and west coast fir.

3. *Norway Pine,* to cover what is known also as "Red Pine."

4. *Hemlock,* to cover Southern or Eastern hemlock; that is, hemlock from all states east of and including Minnesota.

5. *Western Hemlock*, to cover hemlock from the Pacific coast.

6. *Spruce*, to cover Eastern spruce; that is, the spruce timber coming from points east of and including Minnesota.

7. *Western Spruce*, to cover the spruce timber from the Pacific coast.

8. *White Pine*, to cover the timber which has hitherto been known as white pine, from Maine, Michigan, Wisconsin and Minnesota.

9. *Idaho White Pine*, the variety of white pine from western Montana, northern Idaho, and eastern Washington.

10. *Western Pine*, to cover the timber sold as white pine coming from Arizona, California, New Mexico, Colorado, Oregon and Washington. This is the timber sometimes known as "Western Yellow Pine," or "Ponderosa Pine," or "California White Pine," or "Western White Pine."

11. *Western Larch*, to cover the species of larch or tamarack from the Rocky Mountain and Pacific coast regions.

12. *Tamarack*, to cover the timber known as "Tamarack," or "Eastern Tamarack," from States east of and including Minnesota.

13. *Redwood*, to include the California wood usually known by that name.

STANDARD SPECIFICATIONS
FOR
ASPHALT FOR USE IN DAMP-PROOFING AND WATERPROOFING BELOW GROUND LEVEL[1]

Serial Designation: D 40 – 25

These specifications are issued under the fixed designation D 40; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1917; ADOPTED IN AMENDED FORM, 1925.

Scope.

1. These specifications cover asphalt suitable for use as a mopping-coat in damp-proofing, or as a plying-cement in the construction of a membrane system of waterproofing below ground level, under uniformly moderate temperature conditions both during the process of installation and during service.[2]

2. The grade of asphalt covered in these specifications is suitable for damp-proofing and waterproofing foundations, tunnels, subways, etc.

SAMPLING

Sampling.

3. The sampling shall be performed in accordance with the Standard Methods of Sampling Bituminous Materials (Serial Designation: D 140) of the American Society for Testing Materials.[3]

PROPERTIES

Properties.

4. The asphalt shall be homogeneous and free from water. It shall conform to the following requirements:

(*a*) Softening point by the Ring-and-Ball method.....115 to 145° F. (46.1 to 62.7° C.)

(*b*) Penetration at 25° C. (77° F.), 100 g., 5 sec.....50 to 100

(*c*) Flash point (open cup).....not less than 347° F. (175° C.)

(*d*) Loss on heating at 163° C. (325° F.), 50 g., 5 hr.....not more than 2 per cent

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Such asphalt is not intended to be heated above 400° F. (204.4° C.) during its application.

[3] See p. 515.

(*e*) Penetration at 25° C. (77° F.), 100 g., 5 sec., of residue after heating at 163° C. (325° F.) as compared with penetration of asphalt before heating.....not less than 60 per cent

(*f*) Ductility at 25° C. (77° F.).....not less than 30 cm.

(*g*) Insoluble in carbon disulfide.....not more than 5 per cent

METHODS OF TESTING

5. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials: **Methods of Testing.**

(*a*) *Softening Point:* Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (Serial Designation: D 36).[1]

(*b*) *Penetration:* Standard Method of Test for Penetration of Bituminous Materials (Serial Designation: D 5).[2]

(*c*) *Flash Point:* Standard Method of Test for Flash and Fire Points by Means of Open Cup (Serial Designation: D 92).[3]

(*d*) *Loss on Heating:* Standard Method of Test for Loss on Heating of Oil and Asphaltic Compounds (Serial Designation: D 6).[4]

(*e*) *Ductility:* Tentative Method of Test for Ductility of Bituminous Materials (Serial Designation: D 113 – 26 T).[5]

(*f*) *Insoluble in Carbon Disulfide:* Standard Method of Test for the Determination of Bitumen (Serial Designation: D 4).[6]

[1] See p. 496. [2] See p. 493. [3] See p. 404. [4] See p. 489.

[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I, p. 886 (1926); also 1927 Book of A.S.T.M. Tentative Standards, p. 510.

[6] See p. 484.

STANDARD SPECIFICATIONS
FOR
ASPHALT FOR USE IN DAMP-PROOFING AND WATERPROOFING ABOVE GROUND LEVEL[1]

Serial Designation: D 144 – 25

These specifications are issued under the fixed designation D 144; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED IN AMENDED FORM, 1925.

Scope.

1. These specifications cover asphalt suitable for use as a mopping-coat in damp-proofing, or as a plying-cement in the construction of a membrane system of waterproofing above ground level, where not exposed to a temperature exceeding 46° C. (115° F.).[2] It may be used below ground level.

2. The grade of asphalt covered in these specifications is suitable for damp-proofing and waterproofing railroad bridges, tanks, retaining walls, culverts, dams, conduits, etc.

SAMPLING

Sampling.

3. The sampling shall be performed in accordance with the Standard Methods of Sampling Bituminous Materials (Serial Designation: D 140) of the American Society for Testing Materials.[3]

PROPERTIES

Properties.

4. The asphalt shall be homogeneous and free from water. It shall conform to the following requirements:

(*a*) Softening point by the Ring-and-Ball method.....150 to 170° F. (65.5 to 76.7° C.)

(*b*) Penetration at 46.1° C. (115° F.), 50 g., 5 sec.....not more than 100

Penetration at 25° C. (77° F.), 100 g., 5 sec.....25 to 50

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Such asphalt is not intended to be heated above 450° F. (232.2 C.) during its application.

[3] See p. 515.

Penetration at 0° C. (32° F.), 200 g.,
60 sec.....not less than 10
(*c*) Flash point (open cup).....not less than 400° F. (204.4° C.)
(*d*) Loss on heating at 163° C. (325° F.),
50 g., 5 hr.....not more than 1 per cent
(*e*) Penetration at 25° C. (77° F.), 100 g.,
5 sec., of residue after heating at
163° C. (325° F.) as compared with
penetration of asphalt before heating.....not less than 60 per cent
(*f*) Ductility at 25° C. (77° F.) .....not less than 15 cm.
(*g*) Insoluble in carbon disulfide .....not more than 1 per cent

METHODS OF TESTING

Methods of Testing.

5. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Softening Point:* Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (Serial Designation: D 36).[1]

(*b*) *Penetration:* Standard Method of Test for Penetration of Bituminous Materials (Serial Designation: D 5).[2]

(*c*) *Flash Point:* Standard Method of Test for Flash and Fire Points by Means of Open Cup (Serial Designation: D 92).[3]

(*d*) *Loss on Heating:* Standard Method of Test for Loss on Heating of Oil and Asphaltic Compounds (Serial Designation: D 6).[4]

(*e*) *Ductility:* Tentative Method of Test for Ductility of Bituminous Materials (Serial Designation: D 113 – 26 T).[5]

(*f*) *Insoluble in Carbon Disulfide:* Standard Method of Test for the Determination of Bitumen (Serial Designation: D 4).[6]

[1] See p. 496. [2] See p. 493. [3] See p. 404. [4] See p. 489.
[5] *Proceedings* Am. Soc. Testing Mats., Vol. 26, Part I, p. 886 (1926); also 1927 Book of A.S.T.M. Tentative Standards, p. 510.
[6] See p. 484.

STANDARD SPECIFICATIONS
FOR
PRIMER FOR USE WITH ASPHALT IN DAMP-PROOFING AND WATERPROOFING BELOW AND ABOVE GROUND LEVEL[1]

Serial Designation: D 41 – 26

These specifications are issued under the fixed designation D 41; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1917; ADOPTED IN AMENDED FORM, 1925; REVISED, 1926.

Scope.

1. These specifications cover asphaltic primer for use, when specified, with asphalt in damp-proofing and waterproofing below or above ground level, for application to concrete and masonry surfaces.

SAMPLING

Sampling.

2. From each shipment or portion thereof representing primer of the same kind, a number of packages shall be selected at random equivalent to the cube root of the total number of packages in the lot. If the cube root as calculated proves to be a fractional number, it shall be expressed as the next higher whole number. For convenience, the following table is given, showing the number of samples to be selected for shipments of various sizes:

Packages in Shipment	Packages Selected	Packages in Shipment	Packages Selected
2 to 8	2	217 to 343	7
9 to 27	3	344 to 512	8
28 to 64	4	513 to 729	9
65 to 125	5	730 to 1000	10
126 to 216	6	1001 to 1331	11

3. By means of a paddle, the contents of each package selected shall be thoroughly stirred so as to bring all portions thereof into uniform distribution. With an appropriate sampler, not less than 1 pt. of the primer shall be immediately drawn from the center of each package, and transferred to a clean receptacle of suitable size, which in turn shall be kept tightly covered, prior and immediately after said

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

samples are being introduced. After all the pint samples have been added, the contents of the container shall be thoroughly agitated, whereupon 1 qt. of the mixture shall immediately be transferred to a clean quart can provided with a tightly fitting screw cap, which shall be immediately fastened in place. This sample shall be considered as representative of the shipment and shall be transmitted to the laboratory for examination, bearing a suitable tag or other means of identification.

PROPERTIES

Asphaltic Base.

4. The asphaltic base used in preparing the primer shall meet the requirements specified in the Standard Specifications for Asphalt for Use in Damp-proofing and Waterproofing Below Ground Level (Serial Designation: D 40) of the American Society for Testing Materials.[1]

Solvent.

5. The solvent used in preparing the primer shall be a hydrocarbon distillate having an end point on distillation of not above 250° C. (500° F.), of which not more than 20 per cent shall distill under 120° C. (248° F.).

Primer.

6. The primer shall be free from water and shall conform to the following requirements:

(*a*) Sediment.....not more than 1 per cent
(*b*) Asphaltic base by weight.....25 to 35 "

METHODS OF TESTING

Methods of Testing.

7. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials except as specified in Paragraph (*c*):

(*a*) *Distillation:* Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (Serial Designation: D 86).[2]

(*b*) *Sediment:* Standard Method of Test for Water and Sediment in Petroleum Products, by Means of Centrifuge (Serial Designation: D 96).[3]

(*c*) *Weight of Asphaltic Base Present:* Tentative Method of Test for Steam Distillation of Bituminous Protective Coatings (Serial Designation: D 255 – 26 T).[4]

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[1] See p. 704. [2] See p. 378. [3] See p. 437.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I, p. 998 (1926); also 1927 Book of A.S.T.M. Tentative Standards, p. 553.

STANDARD SPECIFICATIONS
FOR
HIGH-CARBON COAL-TAR PITCH FOR USE IN DAMP-PROOFING AND WATERPROOFING BELOW GROUND LEVEL[1]

Serial Designation: D 42 – 25

These specifications are issued under the fixed designation D 42; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1917; ADOPTED IN AMENDED FORM, 1925.

Scope.

1. These specifications cover high-carbon coal-tar pitch suitable for use as a mopping-coat in damp-proofing or as a plying-cement in the construction of a membrane system of waterproofing below ground level, under uniformly moderate temperature conditions both during the process of installation and during service.[2]

2. The grade of high-carbon coal-tar pitch covered by these specifications is suitable for damp-proofing and waterproofing foundations, tunnels, subways, etc.

SAMPLING

Sampling.

3. The sampling shall be performed in accordance with the Standard Methods of Sampling Bituminous Materials (Serial Designation: D 140) of the American Society for Testing Materials.[3]

PROPERTIES

Properties.

4. The coal-tar pitch shall be homogeneous and shall conform to the following requirements:

(*a*) Water.....0.0 per cent

(*b*) Specific gravity at 25° C. (77° F.).....1.24 to 1.34

(*c*) Softening Point[4] (Cube-in-Water Method).....
.....110 to 140°F. (43.3 to 60.0° C.)

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Such coal-tar pitch is not intended to be heated above 300° F. (148.8° C.) during its application.

[3] See p. 515.

[4] The Softening Point (Cube-in-Water Method) specified should have a range of not over 10° F within the limits given in Section 4 (*c*).

(*d*) Distillation Test:

Total distillate, by weight, 0.0 to 300° C. (32 to 572° F.)	not more than 12.0 per cent
Residue, by weight	not less than 88.0 "

(*e*) Specific gravity at 38°/25° C. (100°/77° F.) of total distillate to 300° C. (572° F.) ..... not less than 1.03

(*f*) Ductility at 25° C. (77° F.)..... not less than 50 cm.

(*g*) Insoluble in carbon disulfide..... 20 to 35 per cent.

METHODS OF TESTING

5. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials: Methods of Testing.

(*a*) *Percentage of Water:* Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95).[1]

(*b*) *Specific Gravity:* Standard Method of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements and Soft Tar Pitches (Serial Designation: D 70).[2]

(*c*) *Softening Point:* Standard Method of Test for Softening Point of Tar Products (Cube-in-Water Method) (Serial Designation: D 61).[3]

(*d*) *Distillation:* Tentative Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (Serial Designation: D 20 – 27 T).[4]

(*e*) *Ductility:* Tentative Method of Test for Ductility of Bituminous Materials (Serial Designation: D 113 – 26 T).[5]

(*f*) *Insoluble in Carbon Disulfide:* Standard Method of Test for the Determination of Bitumen (Serial Designation: D 4).[6]

[1] See p. 433. [2] See p. 510. [3] See p. 501.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 505.

[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I, p. 886 (1926); also 1927 Book of A.S.T.M. Tentative Standards, p. 510.

[6] See p. 484.

STANDARD SPECIFICATIONS

FOR

HIGH-CARBON COAL-TAR PITCH FOR USE IN DAMP-PROOFING AND WATERPROOFING ABOVE GROUND LEVEL[1]

Serial Designation: D 145 – 25

These specifications are issued under the fixed designation D 145; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED IN AMENDED FORM, 1925.

Scope.

1. These specifications cover high-carbon coal-tar pitch suitable for use as a mopping-coat in damp-proofing, or as a plying-cement in the construction of a membrane system of waterproofing above ground level, when not exposed to a temperature exceeding 46° C. (115° F.).[2]

2. The grade of high-carbon coal-tar pitch covered in these specifications is suitable for damp-proofing and waterproofing railroad bridges, tanks, retaining walls, culverts, dams, conduits, etc.

SAMPLING

Sampling.

3. The sampling shall be performed in accordance with the Standard Methods of Sampling Bituminous Materials (Serial Designation: D 140) of the American Society for Testing Materials.[3]

PROPERTIES

Properties.

4. The coal-tar pitch shall be homogeneous and shall conform to the following requirements:

(*a*) Water.....0.0 per cent

(*b*) Specific gravity at 25° C. (77° F.).....1.25 to 1.35

(*c*) Softening Point[4] (Cube-in-Water Method).....
.....130 to 155° F. (54.4 to 68.3° C.)

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Such coal-tar pitch is not intended to be heated above 350° F. (176.6° C.) during its application.

[3] See p. 515.

[4] The Softening Point (Cube-in-Water Method) should have a range of not over 10° F. within the limits given in Section 4 (*c*).

(*d*) Distillation Test:

Total distillate, by weight, 0 to 300° C. (32 to 572° F.)	not more than 10.0 per cent
Residue, by weight	not less than 90.0 "

(*e*) Specific gravity at 38°/25° C. (100°/77° F.) of total distillate to 300° C. (572° F.) . . not less than 1.03

(*f*) Ductility at 25° C. (77° F.) . . . . . not less than 20 cm.

(*g*) Insoluble in carbon disulfide . . . . . 22 to 37 per cent.

METHODS OF TESTING

5. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials: Methods of Testing.

(*a*) *Percentage of Water:* Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95).[1]

(*b*) *Specific Gravity:* Standard Method of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements and Soft Tar Pitches (Serial Designation: D 70).[2]

(*c*) *Softening Point:* Standard Method of Test for Softening Point of Tar Products (Cube-in-Water Method) (Serial Designation: D 61).[3]

(*d*) *Distillation:* Tentative Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (Serial Designation: D 20 – 27 T).[4]

(*e*) *Ductility:* Tentative Method of Test for Ductility of Bituminous Materials (Serial Designation: D 113 – 26 T).[5]

(*f*) *Insoluble in Carbon Disulfide:* Standard Method of Test for the Determination of Bitumen (Serial Designation: D 4).[6]

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[1] See p. 433. [2] See p. 510. [3] See p. 501.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 505.

[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I, p. 886 (1926); also 1927 Book of A.S.T.M. Tentative Standards, p. 510.

[6] See p. 484.

STANDARD SPECIFICATIONS
FOR
HIGH-BITUMEN COAL-TAR PITCH FOR USE IN DAMP-PROOFING AND WATERPROOFING BELOW GROUND LEVEL[1]

Serial Designation: D 200 – 25

These specifications are issued under the fixed designation D 200; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1924; ADOPTED, 1925.

Scope.

1. These specifications cover high-bitumen coal-tar pitch suitable for use as a mopping-coat in damp-proofing or as a plying-cement in the construction of a membrane system of waterproofing below ground level, under uniformly moderate temperature conditions both during the process of installation and during service.

2. The grade of high-bitumen coal-tar pitch covered by these specifications is suitable for damp-proofing and waterproofing foundations, tunnels, subways, etc.

SAMPLING

Sampling.

3. The sampling shall be performed in accordance with the Standard Methods of Sampling Bituminous Materials (Serial Designation: D 140) of the American Society for Testing Materials.[2]

PROPERTIES

Properties.

4. The coal-tar pitch shall be homogeneous and shall conform to the following requirements:

(*a*) Water.....0.0 per cent
(*b*) Specific gravity at 25° C. (77° F.).....1.21 to 1.30
(*c*) Softening Point (Cube-in-Water Method).....110 to 140° F. (43.3 to 60° C.)
(*d*) Distillation Test:

Total distillate, by weight, 0.0 to 300° C. (32 to 572° F.).....not more than 15.0 per cent
Residue, by weight.....not less than 85.0 per cent

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] See p. 515.

(*e*) Specific gravity at 38°/25° C. (100°/77° F.) of total distillate to 300° C. (572° F.).....not less than 1.03
(*f*) Ductility at 25° C. (77° F.).....not less than 50 cm.
(*g*) Total Bitumen (Soluble in Carbon Disulfide).....75 to 90 per cent.

METHODS OF TESTING

5. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials: Methods of Testing.

(*a*) *Percentage of Water:* Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95).[1]

(*b*) *Specific Gravity:* Standard Method of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements and Soft Tar Pitches (Serial Designation: D 70).[2]

(*c*) *Softening Point:* Standard Method of Test for Softening Point of Tar Products (Cube-in-Water Method) (Serial Designation: D 61).[3]

(*d*) *Distillation:* Tentative Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (Serial Designation: D 20 – 27 T).[4]

(*e*) *Ductility:* Tentative Method of Test for Ductility of Bituminous Materials (Serial Designation: D 113 – 26 T).[5]

(*f*) *Total Bitumen (Soluble in Carbon Disulfide):* Standard Method of Test for the Determination of Bitumen (Serial Designation: D 4).[6]

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[1] See p. 433. [2] See p. 510. [3] See p. 501.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 505.

[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I, p. 886 (1926); also 1927 Book of A.S.T.M. Tentative Standards, p. 510.

[6] See p. 484.

STANDARD SPECIFICATIONS

FOR

HIGH-BITUMEN COAL-TAR PITCH FOR USE IN DAMP-PROOFING AND WATERPROOFING ABOVE GROUND LEVEL[1]

Serial Designation: D 201 – 25

These specifications are issued under the fixed designation D 201; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1924; ADOPTED, 1925.

Scope.

1. These specifications cover high-bitumen coal-tar pitch suitable for use as a mopping-coat in damp-proofing, or as a plying-cement in the construction of a membrane system of waterproofing above ground level, when not exposed to a temperature exceeding 46° C. (115° F.).

2. The grade of high-bitumen coal-tar pitch covered in these specifications is suitable for damp-proofing and waterproofing railroad bridges, tanks, retaining walls, culverts, dams, conduits, etc.

SAMPLING

Sampling.

3. The sampling shall be performed in accordance with the Standard Methods of Sampling Bituminous Materials (Serial Designation: D 140) of the American Society for Testing Materials.[2]

PROPERTIES

Properties.

4. The coal-tar pitch shall be homogeneous and shall conform to the following requirements:

(*a*) Water.....0.0 per cent

(*b*) Specific gravity at 25° C. (77° F.).....1.22 to 1.31

(*c*) Softening Point (Cube-in-Water Method).....130 to 155° F. (54.4 to 68.3° C.)

(*d*) Distillation Test:

Total distillate, by weight, 0.0 to 300° C. (32 to 572° F.).....not more than 12.0 per cent

Residue, by weight.....not less than 88.0 per cent

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] See p. 515.

(*e*) Specific gravity at 38°/25° C. (100°/77° F.) of total distillate to 300° C. (572° F.).....not less than 1.05
(*f*) Ductility at 25° C. (77° F.).....not less than 20 cm.
(*g*) Total Bitumen (Soluble in Carbon Disulfide).....73 to 88 per cent

METHODS OF TESTING

5. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials: Methods of Testing.

(*a*) *Percentage of Water:* Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95).[1]

(*b*) *Specific Gravity:* Standard Method of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements and Soft Tar Pitches (Serial Designation: D 70).[2]

(*c*) *Softening Point:* Standard Method of Test for Softening Point of Tar Products (Cube-in-Water Method) (Serial Designation: D 61).[3]

(*d*) *Distillation:* Tentative Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (Serial Designation: D 20 – 27 T).[4]

(*e*) *Ductility:* Tentative Method of Test for Ductility of Bituminous Materials (Serial Designation: D 113 – 26 T).[5]

(*f*) *Total Bitumen (Soluble in Carbon Disulfide):* Standard Method of Test for the Determination of Bitumen (Serial Designation: D 4).[6]

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[1] See p. 433. [2] See p. 510. [3] See p. 501.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 505.

[5] *Proceedings*, Am. Soc. Testing Mats., Vol 26, Part I, p. 886 (1926); also 1927 Book of A.S.T.M. Tentative Standards, p. 510.

[6] See p. 484.

STANDARD SPECIFICATIONS
FOR
HIGH-CARBON COAL-TAR PITCH FOR USE IN CONSTRUCTING BUILT-UP ROOFS SURFACED WITH SLAG OR GRAVEL[1]

Serial Designation: D 251 – 27

These specifications are issued under the fixed designation D 251; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1926; ADOPTED, 1927.

Scope.

1. These specifications cover high-carbon coal-tar pitch intended for use as a mopping coat in the construction of built-up roofs surfaced with slag or gravel on inclines not exceeding 3 in. to the foot over boards and 1 in. to the foot over concrete, in connection with the Standard Specifications for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (Serial Designation: D 227) of the American Society for Testing Materials.[2]

SAMPLING

Sampling.

2. The sampling shall be performed in accordance with the Standard Methods of Sampling Bituminous Materials (Serial Designation: D 140) of the American Society for Testing Materials.[3]

PROPERTIES

Properties.

3. The high-carbon coal-tar pitch shall be homogeneous and shall conform to the following requirements:

(*a*) Water..... 0.0 per cent
(*b*) Specific gravity at 25° C. (77° F.)..... 1.25 to 1.34
(*c*) Softening Point (Cube-in-Water Method)..140 to 150° F. (60.0 to 65.5° C.)
(*d*) Distillation Test:
Total distillate, by weight, 0.0 to 300° C. (32 to 572° F.).....not more than 12 per cent
Residue, by weight.....not less than 88 per cent

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] See p. 739. [3] See p. 515.

(*e*) Specific gravity at 38°/25° C. (100°/77° F.) of total distillate to 300° C. (572° F.).....not less than 1.03
(*f*) Ductility at 25° C. (77° F.).....not less than 50 cm.
(*g*) Insoluble in carbon disulfide..... 22 to 35 per cent
(*h*) Ash on incineration.....not more than 0.5 per cent

METHODS OF TESTING

4. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials: **Methods of Testing.**

(*a*) *Percentage of Water:* Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95).[1]
(*b*) *Specific Gravity:* Standard Method of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements and Soft Tar Pitches (Serial Designation: D 70).[2]
(*c*) *Softening Point:* Standard Method of Test for Softening Point of Tar Products (Cube-in-Water Method) (Serial Designation: D 61).[3]
(*d*) *Distillation:* Tentative Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (Serial Designation: D 20 – 27 T).[4]
(*e*) *Ductility:* Tentative Method of Test for Ductility of Bituminous Materials (Serial Designation: D 113 – 26 T).[5]
(*f*) *Insoluble in Carbon Disulfide:* Standard Method of Test for the Determination of Bitumen (Serial Designation: D 4).[6]
(*g*) *Ash:* Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (Serial Designation: D 271).[7]

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[1] See. p. 433. [2] See p. 510. [3] See p. 501.
[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 505.
[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I, p. 886 (1926); also 1927 Book of A.S.T.M. Tentative Standards, p. 510.
[6] See p. 484. [7] See p. 535.

STANDARD SPECIFICATIONS FOR HIGH-BITUMEN COAL-TAR PITCH FOR USE IN CONSTRUCTING BUILT-UP ROOFS SURFACED WITH SLAG OR GRAVEL[1]

Serial Designation: D 252 – 27

These specifications are issued under the fixed designation D 252; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1926; ADOPTED, 1927.

Scope.

1. These specifications cover high-bitumen coal-tar pitch intended for use as a mopping coat in the construction of built-up roofs surfaced with slag or gravel on inclines not exceeding 3 in. to the foot over boards and 1 in. to the foot over concrete, in connection with the Standard Specifications for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (Serial Designation: D 227) of the American Society for Testing Materials.[2]

SAMPLING

Sampling.

2. The sampling shall be performed in accordance with the Standard Methods of Sampling Bituminous Materials (Serial Designation: D 140) of the American Society for Testing Materials.[3]

PROPERTIES

Properties.

3. The high-bitumen coal-tar pitch shall be homogeneous and shall conform to the following requirements:

(*a*) Water.....0.0 per cent
(*b*) Specific gravity at 25° C. (77° F.).....1.22 to 1.32
(*c*) Softening Point (Cube-in-Water Method)...140 to 150° F. (60.0 to 65.5° C.)

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] See p. 739. [3] See p. 515.

(*d*) Distillation Test:

Total distillate, by weight, 0.0 to 300° C. (32 to 572° F.).....not more than 12 per cent
Residue, by weight.....not less than 88 per cent

(*e*) Specific gravity at 38°/25° C. (100°/77° F.) of total distillate to 300° C. (572° F.).....not less than 1.03

(*f*) Ductility at 25° C. (77° F.).....not less than 50 cm.

(*g*) Total bitumen (Soluble in Carbon Disulfide).....78 to 85 per cent

(*h*) Ash on incineration.....not more than 0.5 per cent

METHODS OF TESTING

Methods of Testing.

4. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Percentage of Water:* Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95).[1]

(*b*) *Specific Gravity:* Standard Method of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements and Soft Tar Pitches (Serial Designation: D 70).[2]

(*c*) *Softening Point:* Standard Method of Test for Softening Point of Tar Products (Cube-in-Water Method) (Serial Designation: D 61).[3]

(*d*) *Distillation:* Tentative Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (Serial Designation: D 20 – 27 T).[4]

(*e*) *Ductility:* Tentative Method of Test for Ductility of Bituminous Materials (Serial Designation: D 113 – 26 T).[5]

(*f*) *Insoluble in Carbon Disulfide:* Standard Method of Test for the Determination of Bitumen (Serial Designation: D 4).[6]

(*g*) *Ash:* Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (Serial Designation: D 271).[7]

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[1] See p. 433. [2] See p. 510. [3] See p. 501.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 505.

[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I, p. 886 (1926); also 1927 Book of A.S.T.M. Tentative Standards, p. 510.

[6] See p. 484. [7] See p. 535.

STANDARD SPECIFICATIONS

FOR

CREOSOTE OIL FOR PRIMING COAT WITH COAL-TAR PITCH IN DAMP-PROOFING AND WATERPROOFING BELOW AND ABOVE GROUND LEVEL[1]

Serial Designation: D 43 – 25

These specifications are issued under the fixed designation D 43; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1917; ADOPTED IN AMENDED FORM, 1925.

Scope.

1. These specifications cover creosote oil primer for use, when specified, with coal-tar pitch in damp-proofing and waterproofing below or above ground level, for application to concrete and masonry surfaces.

SAMPLING

Sampling.

2. From each shipment or portion thereof representing creosote oil of the same kind, a number of packages shall be selected at random equivalent to the cube root of the total number of packages in the lot. If the cube root as calculated proves to be a fractional number, it shall be expressed as the next higher whole number. For convenience, the following table is given, showing the number of samples to be selected for shipments of various sizes:

Packages in Shipment.	Packages Selected.	Packages in Shipment.	Packages Selected.
2 to 8	2	217 to 343	7
9 to 27	3	344 to 512	8
28 to 64	4	513 to 729	9
65 to 125	5	730 to 1000	10
126 to 216	6	1001 to 1331	11

3. By means of a paddle, the contents of each package selected shall be thoroughly stirred so as to bring all portions thereof into uniform distribution. With an appropriate sampler, not less than 1 pt. of the creosote oil shall be immediately drawn out from the center of each package and transferred to a clean receptacle of suitable size, which in turn shall be kept tightly covered, prior and immediately after said samples are being introduced. After all the pint

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

samples have been added, the contents of the container shall be thoroughly agitated, whereupon 1 qt. of the mixture shall immediately be transferred to a clean quart can provided with a tightly fitting screw cap, which shall be immediately fastened in place. This sample shall be considered as representative of the shipment and shall be transmitted to the laboratory for examination, bearing a suitable tag or other means of identification.

PROPERTIES

Properties.

4. The creosote oil primer shall conform to the following requirements:

(*a*) Water.....not more than 2 per cent
(*b*) Consistency at 38° C. (100° F.).....entirely fluid
(*c*) Specific gravity at 38°/25° C. (100°/77° F.).....1.00 to 1.06
(*d*) Insoluble in benzol.....not more than 1 per cent
(*e*) Distillation Test:

Total distillate, by weight, under 200° C. (392° F.)	not more than 5 per cent
Total distillate, by weight, under 235° C. (455° F.)	not more than 50 nor less than 30 "
Residue, by weight, above 355° C. (671° F.)	not more than 15 "

(*f*) Specific gravity at 38°/25° C. (100°/77° F.) of the fraction distilling between 235 and 315° C. (455 and 599° F.).....not less than 1.00
(*g*) Consistency at 25° C. (77° F.) of the residue.....soft

METHODS OF TESTING

Methods of Testing.

5. The properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Sampling and Analysis of Creosote Oil (Serial Designation: D 38) of the American Society for Testing Materials.[1]

[1] See p. 683.

STANDARD SPECIFICATIONS
FOR
ASPHALT MASTIC FOR USE IN WATERPROOFING[1]

Serial Designation: D 169 – 25

These specifications are issued under the fixed designation D 169; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED IN AMENDED FORM, 1925.

Scope.

1. (*a*) These specifications cover the materials for asphalt mastic suitable for use in waterproofing, consisting of mastic cake, asphalt cement and mineral aggregate.

(*b*) The grade of asphalt mastic covered by these specifications is suitable for waterproofing bridges, floors, reservoirs, subways, etc.

Properties.

2. This asphalt mastic is a mixture of mastic cake, asphalt cement and mineral aggregate, which at a temperature of approximately 205° C. (400° F.) is sufficiently plastic to be poured into place and compressed with a wooden trowel or equivalent tool into a compact mass.

As used for strictly waterproofing purposes the mastic cake, asphalt cement and mineral aggregate are combined under heat and manual stirring in substantially the following proportions by weight:

Mastic Cake	48 per cent
Asphalt Cement	5 per cent
Mineral Aggregate	47 per cent

Mastic Cake.

3. (*a*) The mastic cake shall conform to the following requirements:

Weight per cake	not more than 75 lb.
Soluble in pure benzol	14 to 18 per cent by weight
Insoluble in pure benzol	82 to 86 per cent by weight

(*b*) The matter soluble in pure benzol shall be asphalt cement, which after recovery from the mastic cake shall conform to the requirements of the Tentative Specifications for Asphalt Cement, 25 to 30 Penetration, for Use in Sheet Asphalt and Asphaltic Concrete Pavements (Serial Designation: D 163 – 23 T) of the American Society for Testing Materials.[2]

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 721 (1923); also 1927 Books of A.S.T.M. Tentative Standards, p. 462.

(*c*) The matter insoluble in pure benzol shall be granular mineral matter which after recovery from the mastic cake shall conform to the following requirements as to grading:

Passing No. 200 sieve	not less than 25 per cent
Passing No. 10 sieve, retained on No. 50 sieve	not more than 25 per cent
Retained on No 10 sieve	not more than 1 per cent

Asphalt Cement.

4. The asphalt cement shall conform to the requirements of the Tentative Specifications for Asphalt Cement, 25 to 30 Penetration, for Use in Sheet Asphalt and Asphaltic Concrete Pavements (Serial Designation: D 163 – 23 T) of the American Society for Testing Materials.[1]

Mineral Aggregate.

5. The mineral aggregate shall be gravel or crushed stone, and sand conforming to the following requirements as to grading:

Passing No. 200 sieve	none
Passing No. 50 sieve, retained on No. 200 sieve	not more than 25 per cent
Passing No. 10 sieve, retained on No. 50 sieve	not less than 25 per cent
Passing No. 4 sieve, retained on No. 10 sieve	not less than 50 per cent
Retained on No. 4 sieve	not more than 10 per cent

Methods of Testing.

6. The properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Bituminous Mastics, Grouts and Like Mixtures (Serial Designation: D 147) of the American Society for Testing Materials.[2]

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 721 (1923); also 1927 Book of A.S.T.M. Tentative Standards, p. 462.

[2] See p. 763.

STANDARD SPECIFICATIONS FOR BITUMINOUS GROUT FOR USE IN WATERPROOFING ABOVE GROUND LEVEL[1]

Serial Designation: D 170 – 25

These specifications are issued under the fixed designation D 170; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED IN AMENDED FORM, 1925.

Scope.

1. (*a*) These specifications cover the materials for bituminous grout suitable for use in waterproofing above ground level, either as a protective covering of membrane systems of waterproofing or for bedding brick or filling the joints or flooding the surface of a brick protective covering.

(*b*) The grade of bituminous grout covered by these specifications is suitable for waterproofing railroad bridges, culverts, subways, etc.

Properties.

2. This bituminous grout is a mixture of substantially 45 parts by weight of bituminous binder and 55 parts by weight of mineral aggregate as coarse as sand, which becomes sufficiently fluid when heated to approximately 205° C. (400° F.), to flow without mechanical manipulation, and which on cooling congeals to a compact mass.

Bituminous Binder.

3. The bituminous binder shall consist of either asphalt binder or coal-tar pitch as follows:

(*a*) The asphalt binder shall conform to the requirements of the Standard Specifications for Asphalt for Use in Damp-proofing and Waterproofing Above Ground Level (Serial Designation: D 144) of the American Society for Testing Materials.[2]

(*b*) The coal-tar pitch shall conform to either of the following specifications of the American Society for Testing Materials:

(1) Standard Specifications for High-Carbon Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level (Serial Designation: D 42);[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] See p. 706. [3] See p. 710.

(2) Standard Specifications for High-Bitumen Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level (Serial Designation: D 200).[1]

Mineral Aggregate.

4. The mineral aggregate shall consist of siliceous sand, all of which will pass a No. 20 sieve, and not more than 5 per cent of which will pass a No. 200 sieve.

Methods of Testing.

5. The properties enumerated in Sections 2 and 4 shall be determined in accordance with the Standard Methods of Testing Bituminous Mastics, Grouts and Like Mixtures (Serial Designation: D 147) of the American Society for Testing Materials.[2]

[1] See p. 714. [2] See p. 763.

STANDARD SPECIFICATIONS FOR BITUMINOUS GROUT FOR USE IN WATERPROOFING BELOW GROUND LEVEL[1]

Serial Designation: D 171 – 25

These specifications are issued under the fixed designation D 171; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED IN AMENDED FORM, 1925.

Scope.

1. (*a*) These specifications cover the materials for bituminous grout suitable for use in waterproofing below ground level, either as a protective covering of membrane systems of waterproofing or for bedding brick or filling the joints or flooding the surface of a brick protective covering.

(*b*) The grade of bituminous grout covered by these specifications is suitable for waterproofing tunnels, subways, etc.

Properties.

2. This bituminous grout is a mixture of substantially 45 parts by weight of bituminous binder and 55 parts by weight of mineral aggregate as coarse as sand, which becomes sufficiently fluid when heated to approximately 149° C. (300° F.), to flow without mechanical manipulation, and which on cooling congeals to a compact mass.

Bituminous Binder.

3. The bituminous binder shall consist of either asphalt binder or coal-tar pitch as follows:

(*a*) The asphalt binder shall conform to the requirements of the Standard Specifications for Asphalt for Use in Damp-proofing and Waterproofing Below Ground Level (Serial Designation: D 40) of the American Society for Testing Materials.[2]

(*b*) The coal-tar pitch shall conform to either of the following specifications of the American Society for Testing Materials:

(1) Standard Specifications for High-Carbon Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level (Serial Designation: D 42).[3]

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] See p. 704. [3] See p. 710.

(2) Standard Specifications for High-Bitumen Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level (Serial Designation: D 200).[1]

Mineral Aggregate.

4. The mineral aggregate shall consist of siliceous sand, all of which will pass a No. 20 sieve, and not more than 5 per cent of which will pass a No. 200 sieve.

Methods of Testing.

5. The properties enumerated in Sections 2 and 4 shall be determined in accordance with the Standard Methods of Testing Bituminous Mastics, Grouts and Like Mixtures (Serial Designation: D 147) of the American Society for Testing Materials.[2]

[1] See p. 714. [2] See p. 763.

STANDARD SPECIFICATIONS FOR WOVEN COTTON FABRICS SATURATED WITH BITUMINOUS SUBSTANCES FOR USE IN WATERPROOFING[1]

Serial Designation: D 173 – 27

These specifications are issued under the fixed designation D 173; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED IN AMENDED FORM, 1925; REVISED, 1927.

Scope.

1. These specifications cover bituminized cotton fabric, composed of woven cotton cloth waterproofed with either asphalt or coal-tar pitch, as specified by the purchaser, for use in the membrane system of waterproofing.

MANUFACTURE

Manufacture.

2. In the process of manufacture, the dry cotton fabric shall be thoroughly and uniformly waterproofed with an asphaltic or coal-tar pitch saturant, at a temperature and speed which will not injure the fabric. This shall be accomplished by passing the fabric through the saturant and then calendering it in the presence of heat, whereupon it shall be cooled and wound into rolls.

PHYSICAL PROPERTIES

Fabricated Bituminized Fabric.

3. The fabricated bituminized cotton fabric shall conform to the following requirements:

(*a*) The width shall be not less than 30 in. nor more than 38 in.

(*b*) The gross weight per roll shall fall within the range of 35 to 80 lb.

(*c*) The wrapping material and mandrel shall not weigh more than 2.5 lb. per roll.

(*d*) The average net weight per square yard shall not be less than 11 oz.

(*e*) The detached comminuted surfacing per 100 sq. ft. shall not weigh more than 1 lb.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

(*f*) The moisture content based on the net weight shall not be more than 1 per cent.

(*g*) The average strength at 70° F. (21.1° C.) measured lengthwise of the roll (that is, in the direction of the warp) shall not be less than 50 lb.[1]

(*h*) The average strength at 70° F. (21.1° C.) measured crosswise of the roll (that is, in the direction of the filling) shall not be less than 40 lb.[1]

(*i*) The pliability at 32° F. (0° C.) shall not be less than 10.

(*j*) The average loss on heating (exclusive of moisture) shall not be more than 4 per cent with asphalt-saturated fabrics.

(*k*) The weight of the saturant contained in the fabricated material shall not be less than twice the weight of the moisture-free fabric in the same area.

Desaturated Fabric.

4. The desaturated woven cotton fabric shall conform to the following requirements:

(*a*) The average dry weight shall not be less than $3\frac{1}{2}$ oz. per sq. yd.

(*b*) The ash based on the dry weight of the fabric shall not be more than 1 per cent.

(*c*) The fabric shall be composed of not less than 100 per cent[2] of cotton fibers.

(*d*) The thread count per inch both in the direction of the warp and in the direction of the filling shall not be more than 36.

WORKMANSHIP AND FINISH

Finished Appearance.

5. The finished material shall be free from visible external defects such as ragged or untrue edges, breaks, rents or cracks.

Degree of Saturation.

6. The meshes of the fabric shall not be completely closed or sealed by the process of saturation, but there shall be sufficient porosity maintained to allow successive moppings of the plying cement to seep through.

Selvage.

7. The selvage shall not measure over $\frac{3}{16}$ in.

Stickiness.

8. The roll shall be capable of being unrolled easily at atmospheric temperatures above 50° F. (10° C.) without sticking together in such a manner as to injure the fabric.

Freedom from Talc.

9. The surface of the fabric shall not be coated or covered with talc or other substance which would tend to interfere with the adhe-

[1] The committee recognizes the advisability of including a requirement relative to the percentage elongation before rupture, but in the absence of any reliable method of test, such requirement cannot be stated at the present time.

[2] This shall not exclude the presence of the customary sizing materials in amounts not exceeding 8 per cent by weight of the moisture-free cotton.

sion between the fabric and the plying cement. The use of silica or wood flour will be permitted.

Finish. 10. The surface shall be uniformly smooth and free from irregularities, folds or knots.

PACKING

Packing. 11. The rolls of treated cotton fabric shall be wound on 2 by 2-in. wooden mandrels extending 2 to 4 in. beyond the ends of the rolls. They shall be securely tied or wrapped in a manner which will prevent unrolling in transit.

SAMPLING AND TESTING

Sampling and Testing. 12. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (Serial Designation: D 146) of the American Society for Testing Materials.[1]

INSPECTION

Inspection. 13. Inspection of material shall be made as agreed upon by the purchaser and seller as part of the purchase contract.

[1] See p. 769.

STANDARD SPECIFICATIONS
FOR
BURLAP SATURATED WITH BITUMINOUS SUBSTANCES FOR USE IN WATERPROOFING[1]

Serial Designation: D 174 – 25

These specifications are issued under the fixed designation D 174; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED IN AMENDED FORM, 1925.

Scope.

1. These specifications cover bituminized jute fabric, composed of burlap waterproofed with either asphalt or coal-tar pitch, as specified by the purchaser, for use in the membrane system of waterproofing.

MANUFACTURE

Material.

2. In the process of manufacture, the dry burlap shall be thoroughly and uniformly waterproofed with an asphaltic or coal-tar pitch saturant at a temperature and speed which will not injure the fabric. This shall be accomplished by passing the fabric through the saturant and then calendering it in the presence of heat, whereupon it shall be cooled and wound into rolls.

PHYSICAL PROPERTIES

Fabricated Bituminized Burlap.

3. The fabricated bituminized burlap shall conform to the following requirements:

(*a*) The width shall be not less than 30 in. nor more than 38 in.

(*b*) The gross weight per roll shall fall within the range of 35 to 80 lb.

(*c*) The wrapping material and mandril shall not weigh more than 2.5 lb. per roll.

(*d*) The average net weight per square yard shall not be less than 20 oz.

(*e*) The detached comminuted surfacing per 100 sq. ft. shall not weigh more than 1 lb.

(*f*) The moisture content based on the net weight shall not be more than 1 per cent.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

(*g*) The average strength at 70° F. (21.1° C.) measured lengthwise of the roll (that is, in the direction of the warp) shall not be less than 70 lb.[1]

(*h*) The average strength at 70° F. (21.1° C.) measured crosswise of the roll (that is, in the direction of the filling) shall not be less than 50 lb.[1]

(*i*) The pliability at 32° F. (0° C.) shall not be less than 10.

(*j*) The average loss on heating (exclusive of moisture) shall not be more than 4 per cent with asphalt-saturated fabrics.

(*k*) The weight of the saturant contained in the fabricated material shall not be less than twice the weight of the moisture-free fabric in the same area.

Desaturated Burlap.

4. The desaturated burlap shall conform to the following requirements:

(*a*) The average dry weight shall not be less than $7\frac{1}{2}$ oz. per sq. yd.

(*b*) The ash based on the dry weight of the fabric shall not be more than 3 per cent.

(*c*) The fabric shall be composed of not less than 100 per cent jute and manila fibers.

WORKMANSHIP AND FINISH

Workmanship and Finish.

5. The finished material shall be free from visible external defects such as ragged or untrue edges, breaks, rents or cracks.

6. The meshes of the fabric shall not be completely closed or sealed by the process of saturation, but there shall be sufficient porosity maintained to allow successive moppings of the plying cement to seep through.

7. The selvage shall not measure over $\frac{3}{16}$ in.

8. The roll shall be capable of being unrolled easily at atmospheric temperatures above 50° F. (10° C.) without sticking together in such a manner as to injure the fabric.

9. The surface of the fabric shall not be coated or covered with talc or other substances which would tend to interfere with the adhesion between the fabric and the plying cement. The use of silica or wood flour will be permitted.

10. The surface shall be uniformly smooth and free from irregularities, folds or knots.

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[1] The committee recognizes the advisability of including a requirement relative to the percentage elongation before rupture, but in the absence of any reliable method of test, such requirement cannot be stated at the present time.

PACKING

Packing.

11. The rolls of treated burlap shall be wound on wooden 2 by 2-in. mandrils extending 2 to 4 in. beyond the ends of the roll. They shall be securely tied or wrapped in a manner which will prevent unrolling in transit.

SAMPLING AND TESTING

Sampling and Testing.

12. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (Serial Designation: D 146) of the American Society for Testing Materials.[1]

INSPECTION

Inspection.

13. Inspection of material shall be made as agreed upon by the purchaser and seller as part of the purchase contract.

[1] See p. 769.

STANDARD SPECIFICATIONS
FOR
ASPHALT-SATURATED ROOFING FELT FOR USE IN WATERPROOFING AND IN CONSTRUCTING BUILT-UP ROOFS[1]

Serial Designation: D 226 – 27

These specifications are issued under the fixed designation D 226; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1925; ADOPTED IN AMENDED FORM, 1927.

Scope.

1. These specifications cover asphalt-saturated felt, either 36 or 32 in. in width, composed of roofing felt saturated, but not coated, with asphalt for use in the membrane system of waterproofing and in the construction of built-up roofs.

MANUFACTURE

Manufacture.

2. In the process of manufacture, a single thickness of dry roofing felt shall be saturated with an asphaltic saturant.

Character of Felt.

3. The felt shall be a roofing-felt produced by "felting" vegetable and animal fibers. The surface of the felt shall be uniformly smooth and, upon splitting or tearing on the bias, shall appear reasonably free from lumps of underbeaten stock (*i.e.*, stock which has not been beaten or shredded into fiber in the process of manufacture) and particles of foreign substances (*i.e.*, fragments of stone, metal, leather, rubber, straw, wood, etc.).

Saturant.

4. The saturant shall be composed of asphaltic materials.

Character of Felt after Saturation.

5. The felt shall be thoroughly and uniformly saturated and shall show no unsaturated spots at any point upon cutting 2-in. strips at random across the entire sheet and splitting them open for their full length.

Surfacing.

6. The surface of the felt shall not be coated or covered with talc or other substance which would tend to interfere with the adhesion between the felt and the plying cement.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

PHYSICAL PROPERTIES

Fabricated Bituminized Felt.

7. The fabricated bituminized felt shall conform to the following requirements:

(*a*) *Width of Roll.*—Either 36 or 32 in., as may be specified, $\pm\frac{1}{4}$ in.

(*b*) *Gross Weight per Roll.*—40 to 80 lb.

(*c*) *Weight of Wrapping and Packing Material per Roll.*—Maximum, $\frac{1}{2}$ lb.

(*d*) *Weight of Material per 100 sq. ft. Exclusive of Packing and Wrapping.*—14 lb. ± 1 lb.

(*e*) *Loss on Heating at 105° C. (221° F.) for 5 hours.*—Maximum, 4 per cent.

(*f*) *Pliability at 77° F. (25° C.).*—At least four strips out of five shall not crack when bent through 180 deg. over a $\frac{1}{16}$-in. mandrel.

(*g*) *Saturant in Moisture-Free Felt.*—Minimum, 140 per cent by weight.

Desaturated Felt.

8. The desaturated felt shall conform to the following requirements:

(*a*) *Weight of Moisture-Free Desaturated Felt.*—Minimum, 5.2 lb. per 100 sq. ft. ("Number" 25).

(*b*) *Ash in Moisture-Free Desaturated Felt.*—Maximum, 10 per cent.

WORKMANSHIP AND FINISH

Freedom from Defects.

9. The finished product shall be free from visible external defects, such as holes, ragged or untrue edges, breaks, cracks, tears, protuberances and indentations.

Stickiness.

10. The rolls shall not crack, or be sticky enough to cause tearing or material damage upon being unrolled at atmospheric temperatures above 50° F. (10° C.).

PACKING

Packing.

11. The rolls of saturated felt need not be wound on cores, but they shall be securely wrapped in a substantial grade of paper of the same width as the fabric. The wrapper shall completely encircle the roll and shall be pasted at the overlap in a manner which will prevent it shifting from position. The ends of the roll need not be covered. As an alternative, the rolls of saturated felt may either be tied with strings or simply pasted at the ends, as may be agreed upon by the purchaser and seller. No roll shall contain more than two pieces,

and there shall not be more than 3 per cent of rolls containing two pieces in any shipment. Each roll shall be plainly marked with the manufacturer's name and brand or as agreed by the purchaser and seller, and shall bear a notation showing the type of saturant.

SAMPLING AND TESTING

Sampling and Testing.

12. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (Serial Designation: D 146) of the American Society for Testing Materials.[1]

INSPECTION

Inspection.

13. Inspection of material shall be made as agreed upon by the purchaser and seller as part of the purchase contract.

[1] See p. 769.

STANDARD SPECIFICATIONS
FOR
COAL-TAR SATURATED ROOFING FELT FOR USE IN WATERPROOFING AND IN CONSTRUCTING BUILT-UP ROOFS[1]

Serial Designation: D 227 – 27

These specifications are issued under the fixed designation D 227; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1925; ADOPTED IN AMENDED FORM, 1927.

Scope.

1. These specifications cover coal-tar saturated felt, either 36 or 32 in. in width, composed of roofing felt saturated, but not coated, with refined coal tar, for use in the membrane system of waterproofing and in the construction of built-up roofs.

MANUFACTURE

Manufacture.

2. In the process of manufacture, a single thickness of dry roofing felt shall be saturated with a coal-tar saturant.

Character of Felt.

3. The felt shall be a roofing-felt produced by "felting" vegetable and animal fibers. The surface of the felt shall be uniformly smooth and, upon splitting or tearing on the bias, shall appear reasonably free from lumps of underbeaten stock (*i.e.*, stock which has not been beaten or shredded into fiber in the process of manufacture) and particles of foreign substances (*i.e.*, fragments of stone, metal, leather, rubber, straw, wood, etc.).

Saturant.

4. The saturant shall be composed of coal tar from which the highly volatile constituents have been removed.

Character of Felt after Saturation.

5. The felt shall be thoroughly and uniformly saturated and shall show no unsaturated spots at any point upon cutting 2-in. strips at random across the entire sheet and splitting them open for their full length.

Surfacing.

6. The surface of the felt shall not be coated or covered with talc or other substance which would tend to interfere with the adhesion between the felt and the plying cement.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

PHYSICAL PROPERTIES

Fabricated Bituminized Felt.

7. The fabricated bituminized felt shall conform to the following requirements:

(*a*) *Width of Roll.*—Either 36 or 32-in., as may be specified, $\pm\frac{1}{4}$ in.

(*b*) *Gross Weight per Roll.*—40 to 80 lb.

(*c*) *Weight of Wrapping and Packing Material per Roll.*—Maximum, $\frac{1}{2}$ lb.

(*d*) *Weight of Material per 100 sq. ft. Exclusive of Packing and Wrapping.*—14 lb. $\pm$1 lb.

(*e*) *Moisture.*—Maximum, 2.5 per cent of the net weight.

(*f*) *Distillate*, per cent by weight to 210° C., calculated on extracted saturant, not more than 2.

Distillate, per cent by weight to 235° C., calculated on extracted saturant, not more than 5.

(*g*) *Pliability at 77° F. (25° C.).*—At least four strips out of five shall not crack when bent through 180 deg. over a $\frac{1}{16}$-in. mandrel.

(*h*) *Saturant in Moisture-Free Felt.*—Minimum, 140 per cent by weight.

Desaturated Felt.

8. The desaturated felt shall conform to the following requirements:

(*a*) *Weight of Moisture-Free Desaturated Felt.*—Minimum, 5.2 lb. per 100 sq. ft. ("Number" 25).

(*b*) *Ash in Moisture-Free Desaturated Felt.*—Maximum, 10 per cent.

WORKMANSHIP AND FINISH

Freedom from Defects.

9. The finished product shall be free from visible external defects, such as holes, ragged or untrue edges, breaks, cracks, tears, protuberances and indentations.

Stickiness.

10. The rolls shall not crack, or be sticky enough to cause tearing or material damage upon being unrolled at atmospheric temperatures above 50° F. (10° C.).

PACKING

Packing.

11. The rolls of saturated felt need not be wound on cores, but they shall be securely wrapped in a substantial grade of paper of the same width as the fabric. The wrapper shall completely encircle the roll and shall be pasted at the overlap in a manner which will prevent it shifting from position. The ends of the roll need not be covered. As an alternative, the rolls of saturated felt may either be tied with strings or simply pasted at the ends, as may be agreed upon by the

purchaser and seller. No roll shall contain more than two pieces, and there shall not be more than 3 per cent of rolls containing two pieces in any shipment. Each roll shall be plainly marked with the manufacturer's name and brand or as agreed by the purchaser and seller, and shall bear a notation showing the type of saturant.

SAMPLING AND TESTING

Sampling and Testing.

12. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (Serial Designation: D 146) of the American Society for Testing Materials.[1]

INSPECTION

Inspection.

13. Inspection of material shall be made as agreed upon by the purchaser and seller as part of the purchase contract.

[1] See p. 769.

STANDARD SPECIFICATIONS
FOR
ASPHALT-SATURATED ASBESTOS FELT FOR USE IN CONSTRUCTING BUILT-UP ROOFS[1]

Serial Designation: D 250 – 27

These specifications are issued under the fixed designation D 250; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1926; ADOPTED IN AMENDED FORM, 1927.

Scope.

1. These specifications cover asphalt-saturated asbestos felt, either 32 or 36 in. in width, composed of asbestos felt saturated, but not coated, with asphalt, for use in the construction of built-up roofs.

MANUFACTURE

Manufacture.

2. In the process of manufacture, a single thickness of asbestos felt shall be saturated with an asphaltic saturant.

Character of Felt.

3. The felt shall be an asbestos felt produced by "felting" at least 85 per cent by weight of asbestos fiber. The surface of the felt shall be uniformly smooth and, upon splitting or tearing on the bias, shall appear reasonably free from lumps or particles of foreign substances.

Saturant.

4. The saturant shall be composed of asphaltic materials.

Character of Felt after Saturation.

5. The felt shall be thoroughly and uniformly saturated and shall show no unsaturated spots at any point upon cutting 2-in. strips at random across the entire sheet and splitting them open for their full length.

Surfacing.

6. The surface of the felt shall not be coated or covered with talc or other substance which would tend to interfere with the adhesion between the felt and the plying cement.

PHYSICAL PROPERTIES

Fabricated Asphalt-Saturated Felt.

7. The fabricated bituminized felt shall conform to the following requirements:

(*a*) *Width of Roll.*—Either 36 or 32 in., as may be specified, $\pm\frac{1}{4}$ in.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

(*b*) *Gross Weight per Roll.*—40 to 80 lb.

(*c*) *Weight of Wrapping and Packing Material per Roll.*—Maximum, $\frac{1}{2}$ lb.

(*d*) *Weight of Material per 100 sq. ft. Exclusive of Packing and Wrapping.*—14 lb. ±1 lb.

(*e*) *Loss on Heating at 105° C. (221° F.) for 5 hours.*—Maximum, 2 per cent.

(*f*) *Pliability at 77° F. (25° C.).*—Shall not crack when bent through 180 deg. over a $\frac{1}{16}$-in. mandrel.

(*g*) *Saturant in Moisture-Free Felt.*—Minimum, 50 per cent by weight.

Desaturated Felt.

8. The desaturated felt shall conform to the following requirements:

(*a*) *Weight of Moisture-Free Desaturated Felt.*—The minimum weight per 100 sq. ft. shall be 9 lb.

(*b*) *Ash in Moisture-Free Desaturated Felt.*—Minimum, 73 per cent.

NOTE.—This presupposes the presence of at least 85 per cent of asbestos fiber carrying 14 per cent water of hydration.

WORKMANSHIP AND FINISH

Freedom from Defects.

9. The finished product shall be free from visible external defects, such as holes, ragged or untrue edges, breaks, cracks, tears, protuberances and indentations.

Stickiness

10. The rolls shall not crack, or be sticky enough to cause tearing or material damage upon being unrolled at atmospheric temperatures above 50° F. (10° C.).

PACKING

Packing

11. The rolls of saturated felt need not be wound on cores, but they shall be securely wrapped in a substantial grade of paper of the same width as the fabric. The wrapper shall completely encircle the roll and shall be pasted at the overlap in a manner which will prevent it shifting from position. The ends of the rolls need not be covered. As an alternative, the rolls of saturated felt may either be tied with strings or simply pasted at the ends, as may be agreed upon by the purchaser and seller. No roll shall contain more than two pieces and there shall not be more than 3 per cent of rolls containing two pieces in any shipment. Each roll shall be plainly marked with the manufacturer's name and brand or as agreed by the purchaser and seller and shall bear a notation showing the type of saturant.

SAMPLING AND TESTING

Sampling and Testing.

12. The material shall be sampled, and the properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (Serial Designation: D 146) of the American Society for Testing Materials.[1]

INSPECTION

Inspection.

13. Inspection of material shall be made as agreed upon by the purchaser and seller as part of the purchase contract.

[1] See p. 769.

STANDARD SPECIFICATIONS FOR ASPHALT ROLL-ROOFING SURFACED WITH POWDERED TALC[1]

Serial Designation: D 224 – 27

These specifications are issued under the fixed designation D 224; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1925; ADOPTED IN AMENDED FORM, 1927.

Scope.

1. These specifications cover smooth-surfaced roll-roofing (medium and heavy weights), either 36 in. or 32 in. in width, composed of roofing felt saturated and coated on both sides with asphalt, and surfaced top and bottom with powdered talc or mica. The medium weight roofing is intended for use on semi-permanent structures and the heavy weight roofing for use on permanent structures.

MANUFACTURE

Manufacture.

2. In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with an asphaltic saturant, then coated on both sides with an asphaltic coating, and finally surfaced on both sides with powdered talc or mica.

Character of Felt.

3. The felt shall be a roofing felt produced by "felting" vegetable and animal fibers. The surface of the felt shall be uniformly smooth, and upon splitting or tearing on the bias, shall appear reasonably free from lumps of underbeaten stock (*i.e.*, stock which has not been beaten or shredded into fiber in the process of manufacture) and particles of foreign substances (*i.e.*, fragments of stone, metal, leather, rubber, straw, wood, etc.).

Character of Saturant and Coatings.

4. The saturant and coatings shall be composed principally of asphaltic materials, and the presence of saponifiable constituents shall be permitted.

Character of Felt after Saturation.

5. The felt shall be thoroughly and uniformly saturated and shall show no unsaturated spots at any point upon cutting 2-in. strips at random across the entire sheet and splitting them open for their full length.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

Character of Mineral Surfacing.

6. The surfacing shall be non-combustible material such as powdered talc or mica.

PHYSICAL PROPERTIES

Physical Properties.

7. The fabricated product shall conform to the following requirements:

(*a*) *Width of Roll.*—Either 36 or 32 in., as may be specified, $\pm\frac{1}{4}$ in.

(*b*) *Area of Roll.*—The average area of the rolls examined shall contain as specified not less than either 108 sq. ft. or 216 sq. ft., which shall be sufficient to cover one or two "squares," respectively.

(*c*) *Weight of Wrapping, Packing, Nails and Cement per "Square."*—Maximum, 4 lb.

NOTE.—Special fixtures may be furnished in lieu of nails and cement, if agreed upon by the purchaser and seller, in which event the above weight may be increased.

(*d*) *Weight of Material per 108 sq. ft. Exclusive of Wrapping, Packing, Loose Mineral Surfacing, Nails and Cement.*—
Medium weight, average, not less than 42 lb., minimum, 40 lb.
Heavy weight, average, not less than 52 lb., minimum, 49 lb.

(*e*) *Pliability at 77° F. (25° C.).*—At least four strips out of five shall not crack on 10-mm. mandrel.

(*f*) *Behavior on Heating to 176° F. (80° C.) for 2 Hours.*—Volatile matter, maximum, 1.5 per cent. There shall be no flowing, sagging, blistering or absorption of the surface coatings.

(*g*) *Saturant in Moisture-free Felt.*—Medium and heavy weights, minimum, 140 per cent by weight.

(*h*) *Weight of Moisture-free Desaturated Felt per 108 sq. ft.*—Medium weight, minimum, 10.8 lb. ("Number" 48).
Heavy weight, minimum, 13.5 lb. ("Number" 60).

(*i*) *Ash in Moisture-free Desaturated Felt.*—Maximum, 10 per cent.

(*j*) *Weight of Mineral Surfacings and Mineral Matter in Coatings per 108 sq. ft.*—Medium and heavy weights, maximum, 2 lb.

NOTE.—Should the weight exceed 2 lb. the material shall nevertheless be acceptable, provided the finished weights per 108 sq. ft. are increased by a like amount over the figures specified in Paragraph (*d*).

WORKMANSHIP AND FINISH

8. The surfaces shall be smooth or veined. Deeply ribbed surfaces shall not be acceptable. **Finished Appearance.**

9. The finished product shall be free from visible external defects, such as holes, ragged or untrue edges, breaks, cracks, tears, protuberances and indentations. **Freedom from Defects.**

10. The coating and mineral surfacing shall be applied uniformly up to the edges of the sheet. **Coatings and Surfacing.**

11. The mineral surfacing shall uniformly cover both sides of the sheet. **Adhesion of Surfacing.**

12. The coating and surfacing shall be of such a nature that the finished product will not crack, or be sticky enough to cause tearing or material damage, upon being unrolled at atmospheric temperatures above 50° F. (10° C.). **Stickiness.**

PACKING

13. The roofing shall be put up in rolls of 108 sq. ft. or 216 sq. ft. as specified in the order. No roll shall contain more than two pieces of roofing, nor shall there be more than 3 per cent of rolls containing two pieces in any shipment. The rolls shall be tightly wound and securely wrapped in a substantial grade of paper. The wrapper shall completely encircle the roll and shall be pasted at the overlap in a manner which will prevent it shifting from position. The ends of the rolls shall be protected with metal caps, cloth or other suitable means. The nails and lap-cement shall be furnished either in each roll, or in bulk as specified in the order. When furnished in bulk, the cloth or metal ends may be omitted. Each roll shall be plainly marked with the manufacturer's name and brand or as agreed upon by the purchaser and seller and shall contain printed directions for laying. Rolls for export shipment shall be packed as agreed upon by the purchaser and seller. **Roll-Roofing.**

14. (*a*) *Nails.*—The gage of the wire shall not be greater than "A. S. & W." No. 10 (0.135 in.) nor thinner than No. 12 (0.105 in.); the head shall not be less than $\frac{3}{8}$ in. in diameter, and not less than 0.025 in. in thickness; the shank shall be between $\frac{3}{4}$ and 1 in. long, with its lower end pointed, and shall be either smooth or barbed, and if the latter, then the barbings shall not increase the diameter of the lower two-thirds more than 0.01 in. The nails shall be zinc coated with not less than $\frac{1}{2}$ oz. nor more than $1\frac{1}{4}$ oz. per sq. ft. of nail surface. Hot-galvanized, electrogalvanized, or sherardized coatings will be accepted. There shall be furnished per "square" not less than an average of 252 nails for 36-in., and not less than an average of 275 nails for 32-in. roofing. **Nails.**

Cement.

(*b*) *Lap-cement.*—The cement shall be composed of bituminous materials dissolved in a volatile solvent, and shall be of such a nature as to firmly bind the laps without injurious effect upon the roofing. The cement shall have a minimum flash point of 71° F. (21.5° C.) when tested by the Tagliabue Closed-Cup Tester. There shall be furnished for each square of 36-in. roofing not less than $\frac{3}{4}$ pint and for 32-in. roofing not less than 1 pint. The addition of 5 to 20 per cent by weight of short-fibered asbestos shall be permitted, in which case the quantity of cement shall be increased by $\frac{1}{4}$ pint per "square."

SAMPLING AND TESTING

Sampling and Testing.

15. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Asphalt Roll-Roofing Surfaced with Fine or Granular Talc, Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles (Serial Designation: D 228) of the American Society for Testing Materials.[1]

INSPECTION

Inspection.

16. Inspection of material shall be made as agreed upon by the purchaser and seller as part of the purchase contract.

[1] See p. 778.

STANDARD SPECIFICATIONS FOR ASPHALT ROLL-ROOFING SURFACED WITH GRANULAR TALC[1]

Serial Designation: D 248 – 27

These specifications are issued under the fixed designation D 248; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1926; ADOPTED, 1927.

Scope.

1. These specifications cover roll-roofing (medium and heavy weights) either 36 or 32 in. in width, composed of roofing felt saturated and coated on both sides with asphalt, and surfaced on the face with granular talc, and on the back with powdered talc or mica. The medium weight is intended for use on semi-permanent structures, and the heavy weight on permanent structures.

MANUFACTURE

Manufacture.

2. In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with an asphaltic saturant, then coated on both sides with an asphaltic coating, and finally surfaced on the weather side with granular talc, and on the reverse side with powdered talc or mica.

Character of Felt.

3. The felt shall be a roofing felt produced by "felting" vegetable and animal fibers. The surface of the felt shall be uniformly smooth and, upon splitting or tearing on the bias, shall appear reasonably free from lumps of underbeaten stock (*i.e.*, stock which has not been beaten or shredded into fiber in the process of manufacture) and particles of foreign substances (*i.e.*, fragments of stone, metal, leather, rubber, straw, wood, etc.).

Character of Saturant and Coatings.

4. The saturant and coatings shall be composed principally of asphaltic materials, and the presence of saponifiable constituents shall be permitted.

Character of Felt after Saturation.

5. The felt shall be thoroughly and uniformly saturated and shall show no unsaturated spots at any point upon cutting 2-in. strips at

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

random across the entire sheet and splitting them open for their full length.

Character of Mineral Surfacing.

6. The surfacing on the weather side of the sheet shall be coarsely crushed talc and of a color as may be agreed upon between the purchaser and seller. It shall be of such a character as to permit the firm adhesion of the larger particles of surfacing and provide a uniform distribution. The under side of the sheet shall be surfaced with a non-combustible material such as powdered mica or talc.

PHYSICAL PROPERTIES

Physical Properties.

7. The fabricated product shall conform to the following requirements:

(*a*) *Width of Roll.*—Either 36 or 32 in., as may be specified, $\pm\frac{1}{4}$ in.

(*b*) *Area of Roll.*—The average area of the rolls examined shall contain, as specified, not less than either 108 sq. ft. or 216 sq. ft., which shall be sufficient to cover one or two "squares" respectively.

(*c*) *Weight of Wrapping, Packing, Nails and Cement per "Square."*—Maximum, 4 lb.

NOTE.—Special fixtures may be furnished in lieu of nails and cement, if agreed upon by the purchaser and seller, in which event the above weight may be increased.

(*d*) *Weight of Material per 108 sq. ft. Exclusive of Wrapping Packing, Loose Mineral Surfacing, Nails and Cement:*
Medium Weight, average, 52 lb.; minimum, 50 lb.
Heavy Weight, average, 62 lb.; minimum, 59 lb.

(*e*) *Pliability at 77° F. (25° C.).*—At least 3 strips out of 5 shall not crack on 10-mm. mandrel.

(*f*) *Behavior on Heating to 176° F. (80° C.) for 2 hours.*—Volatile matter, maximum, 1.5 per cent. There shall be no flowing, sagging, blistering or absorption of the surface coatings.

(*g*) *Saturant in Moisture-Free Felt.*—Medium and Heavy Weights, minimum, 140 per cent by weight.

(*h*) *Weight of Moisture-Free Desaturated Felt per 108 sq. ft.*—Medium weight, minimum, 10.8 lb. ("Number" 48).
Heavy weight, minimum, 13.5 lb. ("Number" 60).

(*i*) *Ash in Moisture-Free Desaturated Felt.*—Maximum, 10 per cent.

(*j*) *Weight of Mineral Surfacings and Mineral Matter in Coatings per 108 sq. ft.*—

Medium and Heavy Weights, maximum, 11 lb.

Medium and Heavy Weights, passing a No. 65 sieve, maximum, 3 lb.

WORKMANSHIP AND FINISH

Finished Appearance.

8. The surfaces shall be smooth or veined. Deeply ribbed surfaces shall not be acceptable.

Freedom from Defects.

9. The finished product shall be free from visible external defects, such as holes, ragged or untrue edges, breaks, cracks, tears, protuberances and indentations.

Coatings and Surfacing.

10. The coating and mineral surfacing shall be applied uniformly up to the edges of the sheet.

Adhesion of Surfacing.

11. The mineral surfacing shall uniformly cover both sides of the sheet.

Stickiness.

12. The coating and surfacing shall be of such a nature that the finished product will not crack, or be sticky enough to cause tearing or material damage upon being unrolled at atmospheric temperatures above 50° F. (10° C.).

PACKING

Roll-Roofing.

13. The roofing shall be put up in rolls of 108 sq. ft. or 216 sq. ft., as specified in the order. No roll shall contain more than two pieces of roofing, nor shall there be more than 3 per cent of rolls containing two pieces in any shipment. The rolls shall be tightly wound and securely wrapped in a substantial grade of paper. The wrapper shall completely encircle the roll and shall be pasted at the overlap in a manner which will prevent it shifting from position. The ends of the rolls shall be protected with metal caps, cloth or other suitable means. The nails and lap-cement shall be furnished either in each roll or in bulk, as specified in the order. When furnished in bulk, the cloth or metal ends may be omitted. Each roll shall be plainly marked with the manufacturer's name and brand or as agreed upon by the purchaser and seller and shall contain printed directions for laying. Rolls for export shipment shall be packed as agreed upon by the purchaser and seller.

Nails.

14. (*a*) *Nails.*—The gage of the wire shall not be greater than "A. S. & W." No. 10 (0.135 in.) nor thinner than No. 12 (0.105 in.); the head shall not be less than $\frac{3}{8}$ in. in diameter, and not less than 0.025 in. in thickness. The shank shall be between $\frac{3}{4}$ and 1 in. long, with its lower end pointed, and shall be either smooth or barbed, and if

the latter, then the barbings shall not increase the diameter of the lower two-thirds more than 0.01 in. The nails shall be zinc coated with not less than $\frac{1}{2}$ oz. nor more than $1\frac{1}{4}$ oz. per sq. ft. of nail surface. Hot-galvanized, electro-galvanized or sherardized coatings will be accepted. There shall be furnished per "square" not less than an average of 252 nails for 36-in., and not less than an average of 275 nails for 32-in. roofing.

Cement. (*b*) *Lap-cement.*—The cement shall be composed of bituminous materials dissolved in a volatile solvent, and shall be of such a nature as to firmly bind the laps without injurious effect upon the roofing. The cement shall have a minimum flash point of 71° F. (21.5° C.) when tested by the Tagliabue Closed-Cup Tester. There shall be furnished for each square of 36-in. roofing not less than $\frac{3}{4}$ pint and for 32-in. roofing not less than 1 pint. The addition of 5 to 20 per cent by weight of short-fibered asbestos shall be permitted, in which case the quantity of cement shall be increased by $\frac{1}{4}$ pint per "square."

SAMPLING AND TESTING

Sampling and Testing. 15. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Asphalt Roll-Roofing Surfaced with Fine or Granular Talc, Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles (Serial Designation: D 228) of the American Society for Testing Materials.[1]

INSPECTION

Inspection. 16. Inspection of material shall be made as agreed upon by the purchaser and seller as part of the purchase contract.

[1] See p. 778.

STANDARD SPECIFICATIONS
FOR
SLATE-SURFACED ASPHALT ROLL-ROOFING AND SLATE-SURFACED ASPHALT SHINGLES[1]

Serial Designation: D 225 – 27

These specifications are issued under the fixed designation D 225; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1925; ADOPTED IN AMENDED FORM, 1927.

Scope.

1. These specifications cover slate-surfaced roll-roofing, either 36 or 32 in. in width, and slate-surfaced composition shingles, composed of roofing-felt saturated and coated on both sides with asphalt and surfaced on the weather side with granulated slate (green, red, black, or such color or combinations of color as may be agreed upon between the purchaser and seller), or similar mineral material to be specified by the purchaser, and on the reverse side with powdered talc or mica to prevent the roofing or shingles adhering together in the package. Such roofing and shingles are intended for use on semi-permanent and permanent buildings.

MANUFACTURE

Manufacture.

2. In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with an asphaltic saturant, then coated on both sides with an asphaltic coating, and finally surfaced on the weather side with granulated slate or similar mineral material to be specified by the purchaser, and on the reverse side with powdered talc or mica.

Character of Felt.

3. The felt shall be a roofing felt produced by "felting" vegetable and animal fibers. The surface of the felt shall be uniformly smooth and, upon splitting or tearing on the bias, shall appear reasonably free from lumps of underbeaten stock (*i.e.*, stock which has not been beaten or shredded into fiber in the process of manufacture) and particles of foreign substances (*i.e.*, fragments of stone, metal, leather, rubber, straw, wood, etc.).

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

Character of Saturant and Coatings.

4. The saturant and coatings shall be composed principally of asphaltic materials, and the presence of saponifiable constituents shall be permitted.

Character of Felt after Saturation.

5. The felt shall be thoroughly and uniformly saturated, and shall show no unsaturated spots at any point upon cutting 2-in. strips at random across the entire sheet and splitting them open for their full length.

Character of Mineral Surfacing.

6. The surfacing on the weather side shall be granular crushed slate or similar mineral material, as may be specified by the purchaser (in either green, red, blue-black or such other color or combinations of color as may be agreed upon between the purchaser and seller), shall be sufficiently free from fines or dust to permit the firm adhesion of the larger particles of the surfacing and provide a uniform distribution. The under side of the sheet shall be surfaced with a non-combustible material such as powered mica or talc.

PHYSICAL PROPERTIES

Physical Properties.

7. The fabricated product shall conform to the following requirements:

(*a*) *Width of Roll; Size and Form of Shingles:* Slate roll-roofing shall be either 36 or 32 in. in width, as may be specified, $\pm\frac{1}{4}$ in.

Shingles shall be of a size and form as may be agreed upon between the purchaser and seller.

(*b*) *Area per Package: Slate-Roll.*—The average area of the rolls examined shall contain not less than 108 sq. ft., which shall be sufficient to cover one "square."

Shingles.—Sufficient material shall be furnished per "square" as may be agreed upon between the purchaser and seller to adequately waterproof, when laid, the surface to which the shingles are applied.

(*c*) *Weight of Wrapping and Packing Material per "Square:" Slate Roll* including nails and lap-cement, maximum, 4 lb.

NOTE.—Special fixtures may be furnished in lieu of nails and cement, if agreed upon by the purchaser and seller, in which event the above weights may be increased.

Shingles (packing only), maximum 12 lb.

(*d*) *Weight of Material per 108 sq. ft. Exclusive of Wrapping, Packing, Loose Mineral Surfacing, Nails and Cement: Slate Roll and Shingles.*—Average, not less than 82 lb., minimum, 80 lb.

(*e*) *Pliability at 77° F. (25° C.): Slate Roll.*—At least four strips out of five shall not crack on 25-mm. mandrel. *Shingles.*—No requirements.

(*f*) *Behavior on Heating to 176° F. (80° C.) for 2 hours.*—Volatile matter, maximum 1.5 per cent. There shall be no flowing, sagging, blistering or absorption of the surface coatings. Granular surfacings shall not slide more than $\frac{1}{16}$ in. when suspended vertically.

(*g*) *Saturant in Moisture-Free Felt:*
Slate Roll, minimum, 140 per cent by weight.
Shingles, minimum, 150 per cent by weight.

(*h*) *Weight of Moisture-Free Desaturated Felt per 108 sq. ft.*—Slate roll and shingles, minimum 10.8 lb. ("Number" 48).

(*i*) *Ash in Moisture-Free Desaturated Felt.*—Maximum, 10 per cent.

(*j*) *Weight of Mineral Surfacings and Mineral Matter in Coating per 108 sq. ft.*—Slate roll and shingles, maximum, 35 lb.

WORKMANSHIP AND FINISH

8. The weather surface shall be uniform in finish and texture. **Finished Appearance.**

9. The finished product shall be free from visible external defects, such as holes, ragged or untrue edges, breaks, cracks, tears, protuberances and indentations. **Freedom from Defects.**

10. The coating and granules shall be applied in a uniformly smooth layer over the entire weather surface, except that slate-roll roofing may be provided with a bare lapping edge, 2 in. wide, along one edge of the sheet, where either the slate alone, or both the slate and coating may be omitted. The under surface of the sheet shall have the asphalt coating and mineral surfacing applied uniformily to the edges. **Distribution of Coatings and Surfacings.**

11. The granular surfacing shall be embedded in the coating in such a manner that when rubbed vigorously with the hand, sufficient surfacing will remain embedded to completely cover the coating. **Adhesion of Surfacing.**

12. For slate roll-roofing, the coating and surfacings shall be of such a nature that the finished product will not crack or stick together so as to cause tearing or damage upon being unrolled at atmospheric temperatures above 50° F. (10° C.). For shingles, the coating and surfacings shall be of such a nature that the finished products will not adhere together in the bundles so as to tear off or loosen the coating when unpacked. **Stickiness.**

PACKING

Roll-Roofing.

13. (*a*) Slate roll-roofing shall be put up in rolls of 108 sq. ft. No rolls shall contain more than two pieces nor shall there be more than 3 per cent of rolls containing two pieces in any shipment. The rolls shall be tightly wound and securely wrapped in a substantial grade of paper. The wrapper shall completely encircle the roll and shall be pasted at the overlap in a manner which will prevent it shifting from position. The ends of the roll shall be protected with metal caps, cloth or other suitable means. The nails and lap-cement shall be furnished either in each roll or in bulk, as specified in the order. When furnished in bulk, the cloth or metal ends may be omitted. Rolls for export shipment shall be packed as agreed upon by the purchaser and seller.

Shingles.

(*b*) Shingles shall be packed in bundles weighing not more than 135 lb. gross. The bundles shall either be packed in cardboard cartons or between wooden or cardboard ends and tied with rope, wire or steel bands so as to make a substantial package, as may be agreed upon by the purchaser and seller. Each roll or package shall be plainly marked with the manufacturer's name and brand or as agreed upon by the purchaser and seller, and shall contain printed directions for laying.

Nails.

14. (*a*) *Nails.*—The gage of the wire shall not be greater than "A. S. & W." No. 10 (0.135 in.) nor thinner than No. 12 (0.105 in.); the head shall be not less than $\frac{3}{8}$ in. in diameter, and not less than 0.025 in. thick; the shank shall be between $\frac{3}{4}$ and 1 in. long for slate-roll roofing, and for shingles 1 in. long when laid on solid sheathing, or $1\frac{1}{2}$ to $1\frac{3}{4}$ in. when laid over old shingles. The lower end of the shank shall be pointed, and either smooth or barbed, and if the latter, then the barbings shall not increase the diameter of the lower two-thirds more than 0.01 in. The nails shall be zinc coated with not less than $\frac{1}{2}$ oz. nor more than $1\frac{1}{4}$ oz. per sq. ft. of nail surface. Hot-galvanized, electrogalvanized, or sherardized coatings will be accepted. There shall be furnished per "square" of slate-roll roofing not less than an average of 252 nails for 36 in., and not less than an average of 275 nails for 32-in. roofing. For shingles, the nails shall be purchased separately, and the number required per "square" will depend upon the size and form of the shingles and shall be agreed upon by the purchaser and seller.

Cement.

(*b*) *Lap-cement* (*for Slate Roll*).—The cement shall be composed of bituminous materials dissolved in a volatile solvent, and shall be of such a nature as to firmly bind the laps without injurious effect upon the roofing. The cement shall have a minimum flash point of

71° F. (21.5° C.) when tested by the Tagliabue Closed-Cup Tester. There shall be furnished for each square of 36-in. roofing not less ¾ pint and for 32-in. roofing not less than 1 pint. The addition of 5 to 20 per cent by weight of short-fibered asbestos shall be permitted, in which case the quantity of cement shall be increased ¼ pint per "square."

SAMPLING AND TESTING

Sampling and Testing.

15. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Asphalt Roll-Roofing Surfaced with Fine or Granular Talc, Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles (Serial Designation: D 228) of the American Society for Testing Materials.[1]

INSPECTION

Inspection.

16. Inspection of material shall be made as agreed upon by the purchaser and seller as part of the purchase contract.

[1] See p. 778.

STANDARD SPECIFICATIONS
FOR
HEAVY WEIGHT SLATE-SURFACED ASPHALT ROLL-ROOFING AND HEAVY WEIGHT SLATE-SURFACED ASPHALT SHINGLES[1]

Serial Designation: D 249 – 27

These specifications are issued under the fixed designation D 249; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1926; ADOPTED, 1927.

Scope.

1. These specifications cover slate-surfaced roll-roofing, either 36 or 32 in. in width, and slate-surfaced composition shingles, composed of roofing-felt saturated and coated on both sides with asphalt and surfaced on the weather side with granulated slate (green, red, black, or such color or combinations of color as may be agreed upon between the purchaser and seller), or similar mineral material to be specified by the purchaser, and on the reverse side with powdered talc or mica to prevent the roofing or shingles adhering together in the package. Such roofing and shingles are intended for use on semi-permanent and permanent buildings.

MANUFACTURE

Manufacture.

2. In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with an asphaltic saturant, then coated on both sides with an asphaltic coating, and finally surfaced on the weather side with granulated slate or similar mineral material to be specified by the purchaser, and on the reverse side with powdered talc or mica.

Character of Felt.

3. The felt shall be a roofing felt produced by "felting" vegetable and animal fibers. The surface of the felt shall be uniformly smooth and, upon splitting or tearing on the bias, shall appear reasonably free from lumps of underbeaten stock (*i.e.*, stock which has not been

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

beaten or shredded into fiber in the process of manufacture) and particles of foreign substances (*i.e.*, fragments of stone, metal, leather, rubber, straw, wood, etc.).

Character of Saturant and Coatings.

4. The saturant and coatings shall be composed principally of asphaltic materials, and the presence of saponifiable constituents shall be permitted.

Character of Felt after Saturation.

5. The felt shall be thoroughly and uniformly saturated, and shall show no unsaturated spots at any point upon cutting 2-in. strips at random across the entire sheet and splitting them open for their full length.

Character of Mineral Surfacing.

6. The surfacing on the weather side shall be granular crushed slate or similar mineral material, as may be specified by the purchaser (in either green, red, blue-black or such other color or combinations of color as may be agreed upon between the purchaser and seller), shall be sufficiently free from fines or dust to permit the firm adhesion of the larger particles of the surfacing and provide a uniform distribution. The under side of the sheet shall be surfaced with a non-combustible material such as powdered mica or talc.

PHYSICAL PROPERTIES

Physical Properties.

7. The fabricated product shall conform to the following requirements:

(*a*) *Width of Roll: Size and Form of Shingles.*—Slate roll-roofing shall be either 36 or 32 in. in width, as may be specified, $\frac{1}{4} \pm$ in.

Shingles shall be of a size and form as may be agreed upon between the purchaser and seller.

(*b*) *Area per Package: Slate-Roll.*—The average area of the rolls examined shall contain not less than 108 sq. ft., which shall be sufficient to cover one "square."

Shingles.—Sufficient material shall be furnished per "square" as may be agreed upon between the purchaser and seller to adequately waterproof, when laid, the surface to which the shingles are applied.

(*c*) *Weight of Wrapping and Packing Material per "Square": Slate Roll* including nails and lap-cement, maximum, 4 lb.

NOTE.—Special fixtures may be furnished in lieu of nails and cement, if agreed upon by the purchaser and seller, in which event the above weight may be increased.

Shingles (packing only), maximum, 12 lb.

(*d*) *Weight of Material per 108 sq. ft. Exclusive of Wrapping, Packing, Loose Mineral Surfacing, Nails and Cement:*

Slate Roll and *Shingles.*—Average, 105 lb., minimum, 100 lb.

(*e*) *Pliability at 77° F. (25° C.): Slate Roll* and *Shingles.*—No requirements.

(*f*) *Behavior on Heating to 176° F. (80° C.) for 2 hours.*—Volatile matter, maximum 1.5 per cent. There shall be no flowing, sagging, blistering or absorption of the surface coatings. Granular surfacings shall not slide more than $\frac{1}{16}$ in. when suspended vertically.

(*g*) *Saturant in Moisture-Free Felt:*
Slate Roll and *Shingles.*—Minimum, 170 per cent by weight.

(*h*) *Weight of Moisture-Free Desaturated Felt per 108 sq. ft.: Slate Roll* and *Shingles.*—Minimum, 15.75 lb. ("Number" 70).

(*i*) *Ash in Moisture-Free Desaturated Felt.*—Maximum, 10 per cent.

(*j*) *Weight of Mineral Surfacings and Mineral Matter in Coating per 108 sq. ft.: Slate Roll* and *Shingles.*—Maximum, 40 lb.

WORKMANSHIP AND FINISH

Finished Appearance. Freedom from Defects.

8. The weather surface shall be uniform in finish and texture.

9. The finished product shall be free from visible external defects, such as holes, ragged or untrue edges, breaks, cracks, tears, protuberances and indentations.

Distribution of Coatings and Surfacing.

10. The coating and granules shall be applied in a uniformly smooth layer over the entire weather surface, except that slate-roll roofing may be provided with a bare lapping edge, 2 in. wide, along one edge of the sheet, where either the slate alone, or both the slate and coating may be omitted. The under surface of the sheet shall have the asphalt coating and mineral surfacing applied uniformly to the edges.

Adhesion of Surfacing.

11. The granular surfacing shall be embedded in the coating in such a manner that when rubbed vigorously with the hand, sufficient surfacing will remain embedded to completely cover the coating.

Stickiness.

12. For slate roll-roofing, the coating and surfacings shall be of such a nature that the finished product will not crack or stick together so as to cause tearing or damage upon being unrolled at atmospheric temperatures above 50° F. (10° C.). For shingles, the coating and surfacings shall be of such a nature that the finished products will

not adhere together in the bundles so as to tear off or loosen the coating when unpacked.

PACKING

13. (*a*) Slate roll-roofing shall be put up in rolls of 108 sq. ft. No rolls shall contain more than two pieces nor shall there be more than 3 per cent of rolls containing two pieces in any shipment. The rolls shall be tightly wound and securely wrapped in a substantial grade of paper. The wrapper shall completely encircle the roll and shall be pasted at the overlap in a manner which will prevent it shifting from position. The ends of the roll shall be protected with metal caps, cloth or other suitable means. The nails and lap-cement shall be furnished either in each roll or in bulk, as specified in the order. When furnished in bulk, the cloth or metal ends may be omitted. Rolls for export shipment shall be packed as agreed upon by the purchaser and seller. **Roll-Roofing.**

(*b*) Shingles shall be packed in bundles weighing not more than 135 lb. gross. The bundles shall either be packed in cardboard cartons or between wooden or cardboard ends and tied with rope, wire or steel bands so as to make a substantial package, as may be agreed upon by the purchaser and seller. Each roll or package shall be plainly marked with the manufacturer's name and brand or as agreed upon by the purchaser and seller, and shall contain printed directions for laying. **Shingles.**

14. (*a*) *Nails.*—The gage of the wire shall not be greater than "A. S. & W." No. 10 (0.135 in.) nor thinner than No. 12 (0.105 in.); the head shall be not less than $\frac{3}{8}$ in. in diameter, and not less than 0.025 in. in thickness; the shank shall be between $\frac{3}{4}$ and 1 in. long for slate-roll-roofing, and for shingles 1 in. long when laid on solid sheathing, or $1\frac{1}{2}$ to $1\frac{3}{4}$ in. when laid over old shingles. The lower end of the shank shall be pointed, and either smooth or barbed, and if the latter, then the barbings shall not increase the diameter of the lower two-thirds more than 0.01 in. The nails shall be zinc coated with not less than $\frac{1}{2}$ oz. nor more than $1\frac{1}{4}$ oz. per sq. ft. of nail surface. Hot-galvanized, electrogalvanized, or sherardized coatings will be accepted. There shall be furnished per "square" of slate-roll-roofing not less than an average of 252 nails for 36-in., and not less than an average of 275 nails for 32-in. roofing. For shingles, the nails shall be purchased separately, and the number required per "square" will depend upon the size and form of the shingles and shall be agreed upon by the purchaser and seller. **Nails.**

Cement.

(*b*) *Lap-Cement* (*for Slate Roll*).—The cement shall be composed of bituminous materials dissolved in a volatile solvent, and shall be of such a nature as to firmly bind the laps without injurious effect upon the roofing. The cement shall have a minimum flash point of 71° F. (21.5° C.) when tested by the Tagliabue Closed-Cup Tester. There shall be furnished for each square of 36-in. roofing not less than $\frac{3}{4}$ pint and for 32-in. roofing not less than 1 pint. The addition of 5 to 20 per cent by weight of short-fibered asbestos shall be permitted, in which case the quantity of cement shall be increased $\frac{1}{4}$ pint per "square."

SAMPLING AND TESTING

Sampling and Testing.

15. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Asphalt Roll-Roofing Surfaced with Fine or Granular Talc, Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles (Serial Designation: D 228) of the American Society for Testing Materials.[1]

INSPECTION

Inspection.

16. Inspection of material shall be made as agreed upon by the purchaser and seller as part of the purchase contract.

[1] See p. 778.

STANDARD METHODS OF TESTING BITUMINOUS MASTICS, GROUTS AND LIKE MIXTURES[1]

Serial Designation: D 147 – 27

These methods are issued under the fixed designation D 147; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED IN AMENDED FORM, 1927.

DEFINITIONS

1. These methods apply to bituminous mastics, grouts and like mixtures, which may be classified and defined as follows: Scope.

Bituminous Grout.—A mixture of bituminous material as a binder and sandy mineral matter as an aggregate, which when heated to a suitable temperature becomes sufficiently fluid to flow into place without mechanical manipulation, and which on cooling congeals to a compact mass.

Asphalt Mastic.—(1) A mixture containing asphaltic material as a binder and graded mineral matter as an aggregate, or (2) pulverized native rock asphalt (to which asphaltic material may have been added); either of which when heated to a suitable temperature may be poured into place but which requires trowelling to form it into a compact mass.

Asphalt Mastic Cake.—A mixture containing asphaltic material as a binder and an aggregate consisting chiefly of calcareous or siliceous dust cast into the form of blocks or "cakes" and adapted for use in preparing asphalt mastic.

PREPARATION OF SAMPLES

2. (*a*) Bituminous grouts shall be heated in an oven or on a hot plate in a pan or other suitable container at the lowest possible temperature to prevent overheating and volatilization, and when sufficiently fluid, shall be thoroughly stirred to insure a uniform sample, whereupon 10 to 30 g. shall be taken for analysis. Preparation of Samples.

(*b*) Asphalt mastics or mastic cake shall be warmed on a hot plate or in a hot oven until soft enough to be broken up or stirred so that a representative sample for analysis may be taken. The amount

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

taken for analysis will depend upon the amount of coarse gravel or stone in the mixture. The larger the gravel or stone, the larger will be the sample required for accuracy. The size of samples to be taken shall be as follows: Where all particles pass a No. 10 sieve, 10 to 30 g.; where 25 per cent of the aggregate is retained on a No. 10 sieve, 50 g.; where 50 per cent of the aggregate is retained on a No. 10 sieve, 100 g.; and where 75 per cent of the aggregate is retained on a No. 10 sieve, 200 g.

NOTE.—Where the properties of the extracted bituminous matter are to be determined, 500 g. shall be taken and extracted with pure benzol as described in Sections 4 and 6 and examined as described in Section 7.

APPARATUS

(*A*) *For Analysis of 10 to 30-g. Samples.*

Apparatus, 10 to 30-g. Samples.

3. In cases where a 10 to 30-g. sample is sufficient, the analysis shall be carried out by means of the glass extractor described in Section 8 of the Standard Methods of Testing Rubber Products (Serial Designation: D 15) of the American Society for Testing Materials.[1]

(*B*) *For Analysis of 50 to 500-g. Samples.*

Apparatus, 50 to 500-g. Samples.

4. The apparatus for analysis of samples containing coarser aggregate shall be the large extractor shown in Fig. 1, consisting of a large brass cylinder, through the bottom of which projects a 16-candle-power incandescent carbon filament bulb to supply heat to the extraction apparatus proper, which is held in the upper portion of the cylinder. This apparatus is composed of a cylindrical brass vessel for holding the solvent, a cylindrical wire basket made of 80-mesh wire cloth suspended in the cylinder, and an inverted conical condenser which serves as a top.

PROCEDURE

(*A*) *Analysis of 10 to 30-g. Samples.*[2]

Procedure.

5. An ordinary Whatman or S. and S. Filter Thimble shall be dried and weighed. The weighed sample shall be placed in the thimble and 40 to 50 cc. of CS_2 poured over the sample. The thimble containing the sample shall be suspended under the condenser by a fine wire bail. The flask shall be cautiously heated by a steam-bath or electric heater just enough to vaporize the solvent. Cold water is circulated through the condenser. The heat evaporates the carbon disulfide in the flask. This condenses upon the condenser and drops back upon

[1] See p. 847.

[2] This method and the method for analysis of larger samples are especially adapted for asphalt mastics, grouts and mastic cake. If the binder is coal-tar pitch, the "free carbon" constituent of the binder will remain with the mineral aggregate. The committee has under consideration a possible separate method for analysis of coal-tar mastics.

the sample through which it filters, thus dissolving out the bitumen which collects in the bottom of the flask.

The extraction should be discontinued when the CS_2 drops colorless from the filter. The time of extraction will depend upon the nature of the bitumen and mineral aggregate in the sample and upon the degree of heat applied, the coldness of the water in the condenser and other factors. In some cases extraction may be complete in one hour, in others four or five hours may be necessary.

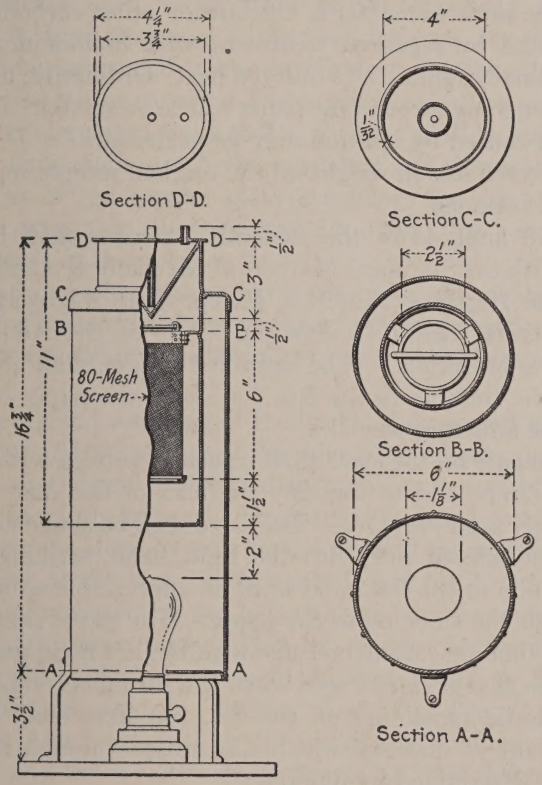

FIG. 1.

When the solvent comes through clear, the filter shall be removed and washed with a fine jet of CS_2 from a washing bottle to wash out any bitumen that may be retained at the top of the paper and to break up any channels that may have been formed by the CS_2 passing through. If the washings show any color, the thimble shall be put back and extraction continued until the solvent again becomes colorless. It shall then be removed, dried carefully, at a low temperature at first to

prevent ignition of the absorbed CS_2, and finally to constant weight at 100° C. (212° F.), cooled and weighed.

The solution in the flask shall be rinsed into a weighed porcelain or silica evaporating dish or crucible and the solvent burned off under a hood. The residue shall be ignited over a flame or in a muffle and the ash weighed and the weight added to that of the mineral matter in the filter paper. This is to correct for the fine mineral matter which will be carried through the paper by the solvent. Should there be a considerable amount of ash recovered in this way, and if it is found that the mineral matter is calcium or other carbonate, it shall be recarbonated by repeated treatment with ammonium carbonate solution and finally ignited at a dull red heat. Ordinarily, however, the mineral matter going through the paper will be so small in amount that the difference caused by ignition may be neglected.

The corrected loss in weight on the original sample represents the percentage of bitumen.

The sieve analysis of the mineral aggregate shall be made in accordance with the Standard Method of Mechanical Analysis of Sand or Other Fine Highway Material, Except Fine Aggregates Used in Cement Concrete (Serial Designation: D 7) of the American Society for Testing Materials.[1] The extracted residue shall now be transferred from the thimble to the No. 200 sieve, the paper being gently rubbed to free adhering particles.

The aggregate on the sieve shall be gently rubbed with the fingers to break up lumps and to free any particles of fine dust that might adhere to larger sand particles. The sieve shall be shaken over a piece of paper from side to side with the right hand, striking it sharply against the palm of the left hand until no appreciable amount of dust comes through the sieve on to the paper. The paper shall be cleared from time to time by raising one side with the left hand and rolling off the siftings, so that it can be seen when the sifting is complete.

The material remaining on the No. 200 sieve shall be weighed and the amount of material which has passed through the No. 200 sieve shall be determined by difference.

This operation shall be repeated upon the coarser sieves in order and the amount passing each sieve and retained on the next finer recorded as percentage of the original sample.

(B) Analysis of 50 to 500-g. Samples.

6. A large filter paper, 12 or 13 in. in diameter, shall be fitted inside the wire basket of the extractor by folding once more than in

[1] See p. 479.

ordinary filtering, or by wrapping it over a form which fits inside the basket (a cylindrical bottle of proper size makes a good form) and placing it inside the basket.

The basket with contained filter paper shall be dried and weighed. The sample shall be weighed and packed in the filter paper in the basket. Care should be taken not to pack all coarse particles in one place and the fine particles in another but to have them mixed together in uniform proportions.

The sample shall be covered with a disk of felt or wad of absorbent cotton to insure even distribution of the dropping solvent, thus preventing it from forming a channel through the sample. The basket shall be suspended in the extractor and 150 to 200 cc. of CS_2 poured over the felt or cotton. The condenser shall be placed over the top and water circulated through it. Current shall be started through the electric lamp underneath the extractor and the extraction carried on exactly as in the smaller glass extractor, but on a larger scale. The time for extraction will vary from 3 to 12 or more hours, depending upon the nature of the sample.

To determine when extraction is complete, the condenser shall be raised and the basket lifted out to observe if drippings are clear. One or two drops caught upon white filter paper should leave but a light stain.

The drying and weighing of the basket, burning off of the solution for correction, and the sifting of the mineral aggregate shall be carried on exactly as in the case of the smaller sample as described in Section 5, except that in sifting mineral aggregates containing coarse stone, it is well to remove the stone by putting the mineral aggregate first through a No. 10 sieve, as the large stone would injure the No. 200 sieve.

The stone removed by the No. 10 sieve may be screened through $\frac{1}{4}$-in., $\frac{1}{2}$-in., and coarser screens in order, as described in the Standard Method of Mechanical Analysis of Broken Stone or Broken Slag, Except Aggregates used in Cement Concretes (Serial Designation: D 18) of the American Society for Testing Materials[1] and the fine material passing the No. 10 sieve through No. 200 and coarser sieves in order, all amounts passing any one screen or sieve and retained on the next finer one being reported as percentage of the original sample.

Thus the ingredients of the mixture may be roughly separated by the use of the proper sized sieves, and examined for physical and chemical characteristics.

NOTE.—The siftings passing the No. 200 sieve will consist largely of the dust or pulverized rock used in the mixture. The material between the No. 100 and

[1] See p. 481.

the No. 10 will consist largely of the sand used in the mixture, with any particles of fine crushed stone within these limits that existed in the original materials. Gravel or broken stone as a rule will be larger than No. 10 or No. 8.

Carbon tetrachloride, benzol, or chloroform may be used instead of carbon disulfide, with the only difference that in the case of non-inflammable solvents, the solvent will have to be evaporated from the solution of bitumen to determine the ash for correction instead of burning off directly. When the solvent is expelled, the bitumen can be ignited for ash.

(*C*) *Recovery and Examination of Extracted Bitumen.*

7. The benzol solution containing the asphalt shall be evaporated to a thick viscous liquid on a steam or water bath, then transferred to a steam oven at 105° C. (221° F.) until its weight is nearly equal to the percentage of asphalt calculated in accordance with Section 5. It shall then be transferred to a hot plate and heated carefully with stirring at a temperature not exceeding 121° C. (250° F.) until its weight is exactly that determined in accordance with Section 6.

(*D*) *Solubility of Aggregate in Hydrochloric Acid.*

8. Where the mastic is to be used for acid-proof floors, tank linings, etc., pulverized silica and other materials insoluble in acid are used in preparing the mixture. The most important chemical property, therefore, is solubility or insolubility in mineral acids. This determination may be carried out in the following manner:

About 2.5 g. of the dust, or of the coarser material, or stone which has been previously pulverized in a mortar shall be dried to constant weight at 325° F. The loss will represent any moisture that may have been retained in the material as well as any absorbed solvent that may not have been expelled while drying the sample after extraction.

A 1-g. sample of the dried material shall be placed in a 250-cc. beaker, covered with a watch glass and 10 cc. of concentrated c. p. hydrochloric acid added, diluted with 50 cc. of distilled water. Any lumps shall be broken up by means of a glass stirring rod. Any effervescence which may occur shall be noted. If considerable effervescing takes place, the sample may be said to be carbonate. If all the sample, or practically all, dissolves, the determination need not be carried farther. If apparently insoluble, it shall be heated carefully, brought to boiling for 15 minutes and filtered through a tared Gooch filter, washing the insoluble residue with hot water. It shall be ignited and weighed and the loss reported as the percentage soluble in hydrochloric acid.

STANDARD METHODS OF TESTING FELTED AND WOVEN FABRICS SATURATED WITH BITUMINOUS SUBSTANCES FOR USE IN WATERPROOFING AND ROOFING[1]

Serial Designation: D 146 – 27

These methods are issued under the fixed designation D 146; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED IN AMENDED FORM, 1927.

Scope.

1. These methods cover the examination of felted or woven fabrics saturated (but not coated) with asphalt or coal-tar products, for use in the "membrane" system of waterproofing.

SAMPLING

Sampling.

2. From each shipment or portion thereof representing a product of the same kind, class and weight, a number of rolls shall be selected at random, equivalent to one-half the cube root of the total number of rolls in the lot, except that in lots of 1000 or less, 5 rolls shall be taken. If the cube root, as calculated, proves to be a fractional number, it shall be expressed as the next higher whole number. For convenience, the following table is given, showing the number of samples to be selected for shipments of various sizes:

Packages in Shipment	Number of Packages Selected	Packages in Shipment	Number of Packages Selected
Up to 1000	5	5 833 to 8 000	10
1001 to 1728	6	8 001 to 10 648	11
1729 to 2744	7	10 649 to 13 842	12
2745 to 4096	8	13 843 to 17 576	13
4097 to 5832	9	17 577 to 21 952	14

EXAMINATION OF FINISHED FABRIC

Width.

3. The width of each roll taken shall be measured to the nearest $\frac{1}{32}$ in. The minimum and maximum width so obtained shall be reported.

Gross Weight per Roll.

4. Each roll taken shall be weighed intact to the nearest $\frac{1}{4}$ lb. and the minimum and maximum gross weight shall be reported.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

Wrapping and Packing Materials.

5. Each roll sampled shall be stripped. All the wrappers and packing material shall be weighed together to the nearest $\frac{1}{4}$ lb. and the average maximum weight thereof per roll shall be reported.

Area per Roll.

6. Each roll shall be unwound, observing the workmanship and finish while so doing. The length and width of each roll shall thereupon be measured to the nearest $\frac{1}{4}$ in. and the square feet of material contained in each roll calculated.

Net Weight per Roll.

7. The rolls shall be rewound, fastened with pieces of light string and then each roll shall be weighed to the nearest $\frac{1}{4}$ lb. In the case of felted fabrics, calculate the weight of each roll in pounds per 100 sq. ft. and report the maximum and minimum, likewise recording the average for the rolls sampled, which shall be regarded as the corresponding weights of the entire shipment. In the case of woven fabrics, calculate the weight of each roll in ounces per square yard, and report the minimum, likewise recording the average for the rolls sampled, which shall be regarded as the corresponding weights of the entire shipment.

Weight per Unit Area.

8. From the rolls examined, select the one whose weight per unit area is nearest the average weight per unit area of the lot. The roll so selected shall be laid flat, the first convolution or two carefully unwound, and with a knife and straight edge the sheet shall be cleanly cut across at right angles to the edges. A section measuring 30 in. in the direction of the roll's length shall be removed. If the material is surfaced with sand or other fine material any detached particles belonging to each 30-in. section shall be retained. The width of each section shall be measured to the nearest $\frac{1}{32}$ in. Each section, together with any detached surfacing, shall be weighed to the nearest gram. The weight in pounds per 100 sq. ft. or the weight in ounces per sq. yd. shall be calculated from the formulas:

$$\text{Pounds per 100 sq. ft.} = 1.0582 \times \frac{\text{Weight of 30-in. section in grams}}{\text{Width of 30-in. section in inches}}$$

$$\text{Ounces per sq. yd.} = 1.5238 \times \frac{\text{Weight of 30-in. section in grams}}{\text{Width of 30-in. section in inches}}$$

This must check within 1 per cent of the average weight per unit area of shipment (Section 7). If the sample fails to do this, then additional samples shall be cut from the same roll, until one is obtained which does, which sample shall be kept for further examination.

NOTE.—As a referee method or in case any dispute arises regarding the properties as may be ascertained from the particular sample selected, a 30-in. sample shall be taken and examined separately from each roll sampled in Section 2.

Detached Comminuted Surfacing.

9. If the material is surfaced with sand or other finely comminuted material, the surfacing shall be swept with a moderately stiff brush from all the 30-in. sections taken. All the material thus removed shall

be caught and weighed to the nearest ounce. From the aggregate areas of all the sections taken, the average weight of detached comminuted surfacing shall be calculated in pounds per 100 sq. ft.

10. The thickness of each specimen shall be measured at 10 equally spaced points, 1 in. from a cut edge, with a micrometer gage having flat bearing surfaces at both contact points of not less than ½ in. in diameter and preferably of the spring-and-dial type. The readings for each roll sampled shall be averaged and the minimum, maximum and average thickness calculated to the nearest 0.001 in. Thickness.

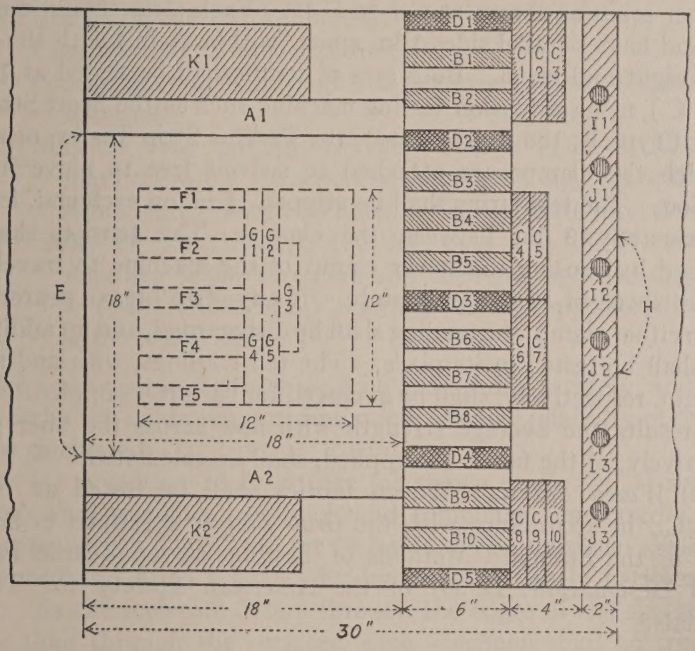

FIG. 1

11. From each specimen, 2-in. strips shall be cut as shown at *A-1* and *A–2* in Fig. 1. The 2-in. strips shall be cut into 1-in. squares. About 50 g., selected at random, shall be accurately weighed and distilled with 100 cc. of a coal-tar distillate of which 5 to 10 per cent boils below 100° C. (212° F.) and of which at least 90 per cent distills up to 180° C. (356° F.) in an apparatus of the form and under the conditions prescribed in the Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95) of the American Society for Testing Materials.[1] The flask containing the solvent and sample shall be heated in a paraffin Moisture.

[1] See p. 433.

bath at a temperature of 302 to 338° F. (150 to 170° C.) until no further water passes over. The distillate shall be allowed to stand in the graduated receiver until the water separates, whereupon the volume of water shall be measured and its weight calculated. From this, the average percentage of moisture in the fabric, as received, shall be calculated.

Strength. 12. (*a*) *Felted Fabrics.*—With a photographic trimmer having a stop attached exactly 1 in. behind the blade, ten strips shall be cut with the fiber grain, as shown at *B-1* to *B-10* (Fig. 1) and ten strips across the fiber grain as shown at *C-1* to *C-10*. Each strip should measure 6 in. and have parallel sides 1 in. apart (within $\frac{1}{32}$ in.) with the edges cut straight and clean. Both sets of strips shall be tested at 70° F. (21.1° C.), using a tension testing machine such as the Scott Strength Tester (Type F, 100 lb. capacity), the Perkins Strip Tester, or equal, in which the clamps are attached to swivels free to move in any direction. The test strips shall be gripped $1\frac{1}{2}$ in. on each end, leaving approximately 3 in. between the clamps. The tension shall be increased by causing the lower clamp of the machine to travel at a uniform speed of 12 in. per minute. If any strip breaks nearer than $\frac{1}{2}$ in. to either clamp, the reading shall be disregarded, and an additional strip shall be tested in its place. The ten readings, with and across the grain, respectively, shall be averaged for each roll sampled. From these results the average strength with and across the fiber grain, respectively, of the fabric as supplied, shall be calculated.

(*b*) *Woven Fabrics*.—Woven fabrics shall be tested at 70° F. (21.1° C.) in accordance with the *Grab Test* as described in Section 12 (*b*) of the Standard Methods of Testing Woven Textile Fabrics (Serial Designation: D 39) of the American Society for Testing Materials.[1]

NOTE.—As a referee method, or in case any dispute arises regarding the strength, the test shall be repeated, with the exception that the fabric before testing shall be exposed at least two hours in an atmosphere of 65 per cent relative humidity at 70° F. (21.1° C.).

Pliability. 13. With the trimmer described in Section 12 (*a*), five 6-in. strips shall be cut with the fiber grain, as shown at *D-1* to *D-5* (Fig. 1) and immersed in water at 77° F. (25° C.) for from 10 to 15 minutes. These strips shall be bent through 180 deg. at a uniform speed, in exactly 2 seconds, around a mandrel, the diameter of which shall be as follows: The first, around a mandrel 25 mm. in diameter; the second, around a mandrel 20 mm. in diameter; the third, around a

[1] See p. 900.

mandrel 15 mm. in diameter; the fourth, around a mandrel 10 mm. in diameter, and the fifth, around a mandrel 5 mm. in diameter. The test may also be made at 32° F. (0° C.). The pliability shall be expressed numerically from 1 to 10 as follows:

(1) Cracks entirely through the sheet on the 25-mm. mandrel.
(2) Cracks part way through the sheet on the 25-mm. mandrel.
(3) Cracks on the 25-mm. mandrel.
(4) Cracks on the 20-mm. mandrel.
(5) Cracks on the 15-mm. mandrel.
(6) Cracks on the 10-mm. mandrel.
(7) Cracks on the 5-mm. mandrel.
(8) Cracks when bent through 180 deg. over a $\frac{1}{16}$-in. (1.588-mm.) mandrel.
(9) May be bent through 180 deg. over a $\frac{1}{16}$-in. (1.588-mm.) mandrel in one direction without cracking, but will crack when bent through 360 deg. in the opposite direction.
(10) May be bent through 180 deg. over a $\frac{1}{16}$-in. (1.588-mm.) mandrel in one direction and then through 360 deg. over a $\frac{1}{16}$-in. (1.588-mm.) mandrel in the opposite direction without cracking.

The readings for each roll shall be averaged separately, and from these results the minimum, maximum and average pliability of the felt shall be calculated.

Water Absorbed.

14. (*a*) A section 18 in. square (as shown at *E*, Fig. 1) shall be cut from each specimen, weighed, and completely immersed in distilled water at 77° F. (25° C.) for 24 hours. The specimen shall then be removed and dried superficially by pressing lightly between two towels. As the moisture enters through the edges of the sheet more rapidly than through the surfaces, each specimen shall be trimmed to exactly 12 in. square, representing four-ninths of the original area, and reweighed rapidly. The increase in weight shall be calculated on the basis of the original test specimen by multiplying by $2\frac{1}{4}$. The percentage increase in weight represents the water absorption. The minimum, maximum and average for the shipment shall be calculated.

(*b*) Five strips shall be cut with the fiber grain as shown at *F 1* to *F 5* (Fig. 1); five other strips shall be cut across the fiber grain as shown at *G 1* to *G 5* (Fig. 1) and the strength shall be redetermined as in Section 12. The decrease in strength in percentage shall be calculated after the specimen has been subjected to water.

Loss on Heating.

15. (*a*) *Asphalt Saturated Products Only.*—From each specimen, 12 by 6-in. strips shall be cut at *K-1* and *K-2* in Fig. 1, care being taken

not to disturb any of the detached surfacing. Each strip shall be weighed and suspended in the center of an air oven maintained at 221° F. (105° C.) ±5° F. (3° C.) with a thin wire fastened through holes punctured near one edge. The thermometer shall be inserted in the oven to such a depth that its bulb will be in line with the center of the specimens. The strips shall be kept in the oven for exactly 5 hours, then cooled and removed carefully, and each specimen weighed. The average loss shall be calculated as a percentage and the average percentage of moisture (ascertained in Section 11) deducted. The final figure shall represent the average loss on heating, exclusive of moisture.

Extraction of Saturant.

(*b*) *Coal-Tar Saturated Felt Only.*—From the rolls making up the sample, strips 6 in. wide shall be cut across the sheet. Sufficient of these shall be taken to make up a sample of 250 g. ±5.0 g. These strips then shall be rolled up and placed in the wire basket of the extraction apparatus described in the Standard Methods of Testing Bituminous Mastics, Grouts and Like Mixtures (Serial Designation: D 147) of the American Society for Testing Materials[1] or other extraction apparatus of suitable construction. The sample shall then be covered with a disk of soft filter paper to insure an even distribution of the solvent. The extraction shall be carried out as described in Methods D 147.

Recovery of Saturant from CS_2 Extract.

The CS_2 extract containing the bitumen shall be transferred to a 500-cc., short-neck, round-bottom flask. The flask shall be equipped with a Hempel column, 16 cm. in length, 15 mm. in inside diameter and filled to a depth of 5 cm. with glass beads. The Hempel column shall be connected with a water cooled condenser. A −2 to +80° C. thermometer as described in the Standard Method of Float Test for Bituminous Materials (Serial Designation: D 139) of the American Society for Testing Materials[2] shall be placed in the Hempel column so that the top of the bulb shall be opposite the bottom of the tubulure. Distillation for the removal of CS_2 shall be continued by means of a steam bath until drops cease to fall from the condenser. The distillation then shall be carried out over a small flame, heating carefully and observing the thermometer until the distillation ceases and the temperature of the vapor as indicated by the thermometer shows no further rise on slightly increasing the flame.

Distillation of Saturant.

When the contents of the flask is sufficiently cool to be poured without appreciable vaporizing, 100 g. ±0.1 g. shall be distilled in accordance with the Tentative Method of Test for Distillation of

[1] See p. 763. [2] See p. 506.

Creosote Oil (Serial Designation: D 246 – 27 T) of the American Society for Testing Materials.[1]

Fractions shall be taken at 210° C. and 235° C. and the distillation stopped at that point. The per cent of distillate by weight at each temperature shall be calculated on the basis of the weight of sample taken for distillation.

DESATURATING THE FABRIC

Desaturated Fabric.

16. From each roll sampled, a 2-in. strip (within $\frac{1}{32}$ in.) shall be cut across the specimen as shown at *H* (Fig. 1). Each strip shall be extracted with carbon disulfide[2] in an extractor described in Section 4 of the Standard Methods of Testing Bituminous Mastics, Grouts and Like Mixtures (Serial Designation: D 147) of the American Society for Testing Materials,[3] the extraction being continued for several hours after the drippings have become colorless. The desaturated fabric shall be removed and heated in a ventilated oven at 225° F. (107.1° C.), cooled in a desiccator, any adhering comminuted surfacing being brushed off and retained, and weighed as rapidly as possible. The heating shall be repeated until the weight of the fabric remains constant as determined by two consecutive weighings taken not less than ten minutes apart which shall show a further loss of not more than 0.1 per cent. This will give the weight of the fabric in the moisture-free state. Where a coal-tar pitch saturant has been used, the moisture-free weight of the desaturated fabric shall be corrected for the carbonaceous matter retained mechanically in its interstices, by means of the method described in Section 17.

Retained Carbonaceous Matter.

17. The following colorimetric method shall be used to ascertain the carbonaceous matter derived from a coal-tar pitch saturant and retained by the desaturated fabric:

(*a*) About 15 g. of an unsaturated fabric of the same general character as the one under examination, shall be macerated by boiling in water, disintegrating with a rotary egg-beater and picking the fibers apart with needles. The fibers shall be filtered through fine cloth and dried at a temperature of 225° F. (107.1° C.) to constant weight. One gram of the fibers shall be accurately weighed into a flask and diluted to exactly 100 cc. with distilled water at room temperature. About 50 g. of glass beads shall be added and the contents of the flask shaken vigorously until the fibers are reduced to a homogeneous pulp in uniform suspension.

(*b*) A distilled coal-tar, having approximately 10 to 25 per cent

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 531.

[2] In this test, chloroform may be used as an equivalent solvent.

[3] See p. 763.

of insoluble carbonaceous matter, shall be procured and the carbonaceous matter extracted with benzol until it is free from soluble matter; then dried at a temperature of 225° F. (107.1° C.) to constant weight. One gram of the purified carbonaceous matter shall be accurately weighed and diluted to exactly 100 cc. at room temperature with a starch solution of a consistency sufficient to carry the carbonaceous matter into temporary suspension. (A 12.5-per-cent solution is recommended.)

(*c*) The liquid carrying the fibers, obtained as described in Paragraph (*a*), shall be titrated with the suspension of carbonaceous matter, obtained as described in Paragraph (*b*), and from time to time a field prepared from a drop of the well agitated mixture shall be examined under a microscope at 100 diameters magnification until the color exactly matches a field prepared from the desaturated fabric under examination (obtained as described in Section 16), when both are viewed side by side under parallel conditions. The end-point is fairly sharply defined. The burette reading gives directly the percentage of carbonaceous matter adhering to the moisture-free fabric under examination.

(*d*) The weight of moisture-free fabric ascertained in Section 16 shall be corrected by deducting the weight of adhering carbonaceous matter.

Total Comminuted Surfacing.

18. The total comminuted surfacing plus any filler present in the bituminous saturant represents the sum of the detached comminuted surfacing (Section 9), the amount brushed off from the surface of the desaturated fabric (Section 16), plus the amount recovered upon evaporating and igniting the bituminous extract obtained in accordance with Section 16.

Bituminous Saturant.

19. The weight of bituminous saturant represents the difference between the weight of the original fabric (Section 7), and the sum of the weights of the moisture-free desaturated fabric (Sections 16 and 17), plus moisture (Section 11), plus the total comminuted surfacing and filler present (Section 18).

EXAMINATION OF THE EXTRACTED FABRIC

(*A*) *Woven Fabrics*

Weight.

20. Woven fabrics shall be tested for weight and number of threads per in. in accordance with Sections 7 to 9, inclusive, of the Standard Methods of Testing Woven Textile Fabrics (Serial Designation: D 39) of the American Society for Testing Materials.[1]

[1] See p. 900.

(*B*) *Felted Fabrics*

21. The weight in pounds per 480 sq. ft. of the desaturated felt (corrected for any carbonaceous matter present) for each specimen examined, shall be calculated separately from the formulas: Weight.

$$\text{"Number"} = \frac{\text{Weight of specimen in grams}}{\text{Area of specimen in square centimeters}} \times 983$$

or

$$\text{"Number"} = \frac{\text{Weight of specimen in grams}}{\text{Area of specimen in square inches}} \times 152$$

This represents the "number" of the moisture-free felt on the felt makers' scale. The minimum, maximum and average "number" of the felt in its moisture-free state shall be calculated. This may be corrected to the condition in which it actually existed in the material under examination, by adding thereto the percentage of moisture as determined in Section 11.

22. A representative sample shall be secured by cutting from each strip of desaturated felt a piece about $\frac{1}{2}$ in. in diameter as shown at *I-1*, *I-2*, and *I-3* (Fig. 1). About 25 g. selected at random from all the specimens sampled in this manner shall be accurately weighed, and incinerated in a weighed porcelain or quartz crucible either over an open flame or in a muffle, until all the carbon is consumed. A few drops of ammonium carbonate solution shall be added, the sample ignited gently, and weighed. The percentage of ash shall be calculated on the basis of the moisture-free felt. Ash.

NOTE.—As a referee method, or in case greater accuracy is desired, the three portions taken from each specimen roll shall be weighed and ignited separately. The minimum, maximum and average of ash present shall be calculated on the basis of the moisture-free felt.

STANDARD METHODS OF TESTING ASPHALT ROLL-ROOFING SURFACED WITH FINE OR GRANULAR TALC, SLATE-SURFACED ASPHALT ROLL-ROOFING AND SLATE-SURFACED ASPHALT SHINGLES[1]

Serial Designation: D 228 – 27

These methods are issued under the fixed designation D 228; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1925; ADOPTED IN AMENDED FORM, 1927.

Scope.

1. These methods cover the examination of roofings and shingles composed of roofing-felt, saturated and coated on both sides with asphalt, and surfaced on the top with either powdered or granulated mineral matter, and on the under side with powdered mineral matter.

SAMPLING

Sampling.

2. From each shipment or portion thereof representing a product of the same kind, class and weight, a number of rolls or bundles shall be selected at random, equivalent to one-half the cube root of the total number of rolls or bundles included in the lot, except that in lots of 1000 or less, five rolls or bundles shall be taken. If the cube root, as calculated, proves to be a fractional number, it shall be expressed as the nearest higher whole number. For convenience, the following table is given, showing the number of samples to be selected from shipments of various sizes:

Packages in Shipment	Number of Packages Selected	Packages in Shipment	Number of Packages Selected
Up to 1000	5	5 833 to 8 000	10
1001 to 1728	6	8 001 to 10 648	11
1729 to 2744	7	10 649 to 13 824	12
2745 to 4096	8	13 825 to 17 576	13
4097 to 5832	9	17 577 to 21 952	14

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

DETERMINATION OF SIZE AND WEIGHT

(*A*) *Smooth-roll and Slate-roll Roofing*

Stripping.

3. Each roll selected shall be stripped. The wrappers, packing material, nails and cement taken from each roll shall be weighed together to the nearest $\frac{1}{4}$ lb. and the maximum weight thereof per roll shall be recorded. The quantity and character of nails and cement shall be recorded.

Workmanship and Finish.

4. Each roll shall be unwound, discarding any detached surfacing that may fall off in so doing, and the workmanship and finish of the roofing observed.

Area.

5. The length and width of each roll shall be measured to the nearest $\frac{1}{4}$ in., and the square feet of material contained in each roll calculated. From this, the average area of the rolls examined shall be calculated.

Minimum Weight.

6. The rolls shall be rewound, fastened with pieces of light string and then each roll shall be weighed to the nearest $\frac{1}{4}$ lb. The weight of roofing per 108 sq. ft. contained in each roll shall be calculated and the minimum weight per 108 sq. ft. recorded. This shall be regarded as the minimum weight per 108 sq. ft. of the entire shipment.

Average Weight.

7. The average weight per 108 sq. ft. for the rolls examined shall be calculated. This shall be regarded as the average weight per 108 sq. ft. of the entire shipment.

Weight per 108 sq. ft.

8. From the rolls examined, the one whose weight per 108 sq. ft. is nearest the average weight of the lot shall be selected. The first convolution or two shall be unrolled, and with a knife and straight edge, the sheet shall be cut across at right angles to the edges. A section measuring exactly 30 in. in the direction of the roll's length shall then be removed. The width of this section shall be measured to the nearest $\frac{1}{32}$ in. and weighed with an accuracy of $\frac{1}{4}$ oz. The weight in pounds per 108 sq. ft. shall then be calculated from the following formula:

$$\text{Pounds per 108 sq. ft.} = \frac{\text{Weight of 30-in. section in ounces}}{\text{Width of 30-in. section in inches}} \times 32.4$$

This must check within 1 per cent of the average weight per 108 sq. ft. of the shipment (Section 7). If this sample fails to do this, then additional samples shall be cut from the same roll, until one is obtained which does, which sample shall be kept for further examination.

NOTE.—As a referee method, or in case any dispute arises regarding the properties as may be ascertained from the particular sample selected, a 30-in. sample shall be taken and examined separately from each roll sampled in Section 2.

(*B*) *Slate-surfaced Shingles*

Stripping.

9. Each bundle selected shall be stripped. The packing material shall be weighed to the nearest $\frac{1}{4}$ lb., recording the weight separately for each bundle. The maximum weight of packing material per "square" shall be recorded.

Workmanship and Finish.

10. The shingles in each bundle, a, shall be counted, discarding any detached surfacing that may fall off in so doing. The workmanship and finish of the shingles shall be observed.

Area.

11. The shingles contained in each bundle shall be weighed to the nearest $\frac{1}{4}$ lb. (b). The dimensions of the shingles shall be accurately measured and the net area of material in each shingle calculated in square feet (c).

Minimum Weight.

12. The area in square feet of material contained in each bundle ($a \times c$) shall be calculated. The weight of roofing material per 108 sq. ft. contained in each bundle $\left(\frac{b}{a \times c} \times 108\right)$ shall be calculated, and the minimum weight per 108 sq. ft. recorded. This shall be regarded as the minimum weight per 108 sq. ft. of the entire shipment.

Average Weight.

13. The average weight (d) per 108 sq. ft. for the bundles examined shall be calculated. This shall be regarded as the average weight per 108 sq. ft. of the entire shipment. Also, the average weight per shingle $\left(\frac{c \times d}{108}\right)$ shall be calculated.

Selective Sampling.

14. From the bundles selected, a definite number of representative shingles, whose weight shall fall within 1 per cent of the average weight ascertained in Section 13 shall be taken for further consideration. The number of shingles so selected shall represent as closely as possible 6 sq. ft. of material, and shall be selected as far as possible from different bundles.

NOTE.—As a referee method, or in case any dispute arises regarding the properties as may be ascertained from the particular sample selected, then a similar sample shall be taken and examined separately from each bundle of shingles sampled in Section 2.

DETERMINATION OF PLIABILITY OF SMOOTH-ROLL ROOFING AND SLATE-ROLL ROOFING

Pliability.

15. With a photographic trimmer five strips shall be cut from the sample of roofing, each 1 in. in width and 6 in. in length, in the direction of the fiber grain, and immersed in water at 77° F. (25° C.) for 10 to 15 minutes. These strips shall be removed from the water and

immediately bent through 180 deg. at uniform speed, in exactly 2 seconds, over a mandrel of the following size:

Smooth-roll Roofing—At least 3 of the 5 strips shall not crack over a mandrel 10 mm. in diameter.

Slate-roll Roofing—At least 3 of the 5 strips shall not crack on a mandrel 25 mm. in diameter.

DETERMINATION OF BEHAVIOR ON HEATING

Behavior on Heating.

16. Two strips shall be cut from the sample of roofing or shingles, each approximately 8 by 8 in. They shall be weighed and suspended vertically in the center of an air-oven maintained at 176° F. (80° C.) ±5°F. (3° C.) with a thin wire fastened through holes punctured near one edge. The thermometer shall be inserted in the oven to such a depth that its bulb will be in line with the center of the specimens. The strips shall be maintained at the prescribed temperature for exactly two hours, then cooled, removed carefully and each specimen weighed. The average loss shall be calculated as a percentage. Any change in appearance of the specimen shall be recorded, such as flowing, sagging, blistering or absorption of the surface coatings; also sliding of granular surfacing.

DETERMINATION OF SATURANT IN MOISTURE-FREE FELT

Percentage of Saturation.

17. Two strips shall be cut from the sample of roofing or shingles, each approximately 2 in. in width and 8 in. in length. They shall be warmed in an oven at a temperature of approximately 176° F. (80° C.). Then with a knife, the front and back coatings shall be peeled off, care being taken to remove as little as possible of the saturated felt, and to assure that all of the coatings and surfacing are stripped from the felt. The saturated felt so obtained shall be weighed. It shall be extracted with benzol, chloroform, carbon tetrachloride or carbon disulfide, in a suitable extractor and the extraction continued until the drippings have become colorless. The desaturated felt shall be removed from the extractor, placed in a tared vessel, heated in a ventilated oven at 225° F. (107° C.) for 30 minutes, cooled in a desiccator, and the felt finally weighed as rapidly as possible. The heating shall be repeated until the weight of the felt remains constant as determined by two consecutive weighings taken not less than 10 minutes apart, which shall show a further loss of not more than 0.1 per cent. From the weight so obtained and the original weight, the percentage of saturation carried by the moisture-free felt shall be computed.

DETERMINATION OF WEIGHT OF MOISTURE-FREE FELT

Weight of Moisture-Free Felt.

18. Two strips shall be cut from the sample of roofing or shingles, each approximately 2 in. in width and 8 in. in length. Each of these shall be accurately weighed in grams and the length and width measured to within 1 mm. or $\frac{1}{32}$ in. It shall be extracted with carbon disulfide,[1] in a suitable extractor and the extraction continued until the drippings have become colorless. The desaturated felt shall be removed from the extractor, being careful not to lose any adhering mineral matter, placed in a tared vessel, heated in a ventilated oven, at 225° F. (107° C.) for 30 minutes, cooled in a desiccator, any adhering surfacing material brushed off and retained and the felt finally weighed as rapidly as possible. The heating shall be repeated until the weight of the felt remains constant as determined by two consecutive weighings taken not less than 10 minutes apart, which shall show a further loss of not more than 0.1 per cent. The carbon disulfide extract and accompanying mineral matter shall be retained for further examination. From the weights so obtained and the respective areas of the strips of desaturated felt, the weight of moisture-free felt shall be computed in pounds per 108 sq. ft., from the following formulas and the results averaged:

$$\text{Pounds per 108 sq. ft.} = \frac{\text{Weight of moisture-free felt in grams}}{\text{Area of specimen in square centimeters}} \times 221.2$$

or

$$\text{Pounds per 108 sq. ft.} = \frac{\text{Weight of moisture-free felt in grams}}{\text{Area of specimen in square inches}} \times 34.3$$

DETERMINATION OF ASH IN MOISTURE-FREE FELT

Ash.

19. The strips of desaturated felt obtained as described in Section 18 shall be cut into squares about 2 cm. on each side and thoroughly mixed. Pieces amounting to about 10 g. shall be selected at random and dried at 225° F. (107° C.) for 30 minutes. They shall be cooled in a desiccator, accurately weighed and then incinerated in a weighed crucible either over an open flame or in a muffle until all the carbon is consumed. A few drops of ammonium carbonate solution shall then be added, the sample ignited gently and weighed. The percentage of ash shall be calculated on the basis of the moisture-free felt.

DETERMINATION OF MINERAL SURFACING AND MINERAL MATTER IN COATINGS

Mineral Matter.

20. (*a*) The slate and other mineral matter recovered from the extraction process described in Section 18 shall be combined with the mineral matter removed from the surface of the desaturated felt and

[1] In this test, chloroform may be used as an equivalent solvent.

that resulting from the evaporation and ignition of the carbon disulfide extract. The mineral matter shall be dried in air free from the odor of carbon disulfide, then heated in an oven for 30 minutes at 225° F. (107° C.), and finally cooled in a desiccator and weighed. From the combined weight of the specimens taken in Section 18, the total "mineral surfacing and mineral matter in the coatings" shall be calculated in pounds per 108 sq. ft.

(*b*) For asphalt roll-roofing surfaced with granular talc, the mineral matter recovered as described in Paragraph (*a*) shall be washed through a No. 65 copper-wire sieve with three washings of water of 100 cc. each. The total aqueous suspension of mineral matter which passes through the sieve shall then be filtered through a weighed Gooch crucible. It shall then be dried at 225° F. (107° C.) and reweighed. The difference in weight represents the mineral matter passing a No. 65 sieve which shall be converted into pounds per 108 sq. ft.

STANDARD DEFINITION OF THE TERM SLATE[1]

Serial Designation: D 247 – 27

This definition is issued under the fixed designation D 247; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1926; ADOPTED IN AMENDED FORM, 1927.

Slate.—A microgranular crystallin stone derived from argillaceous sediments by regional metamorphism, and characterized by a perfect cleavage entirely independent of original bedding, which cleavage has been induced by pressure within the earth.

NOTE 1.—The essential mineral constituents are white mica (chiefly sericite) and quartz. Prominent accessory constituents are black mica (biotite), chlorite and hematite. Minor accessory constituents are carbonates, magnetite, apatite clay, andalusite, barite, rutile, purite, graphite, feldspar, zircon, tourmaline and carbonaceous matter.

NOTE 2.—Igneous slates because of their rare occurrence and insignificant commercial importance are not covered in this definition.

[1] Under the standardization procedure of the Society, this definition is under the jurisdiction of the A.S.T.M. Committee D-16 on Slate.

STANDARD METHODS OF TESTING MOLDED INSULATING MATERIALS[1]

Serial Designation: D 48 – 27

These methods are issued under the fixed designation D 48; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1917; ADOPTED IN AMENDED FORM, 1922; REVISED, 1924, 1927.

Scope.

1. These tests are intended to apply to all solid insulating materials that are formed in molds by the application of pressure, either with or without heat.

TENSILE STRENGTH

Apparatus.

2. Any standard testing machine may be used provided the error in the loading range does not exceed 1 per cent. Special clips (see Fig. 1) of hardened steel shall be used, hung from links held in the jaws of the machine, so that the pull is central at all times, to avoid any transverse strain.

Specimen.

3. The standard test specimen shown in Fig. 1 shall be used for the tension tests. It shall be molded to the dimensions shown in Fig. 1.

Method

4. (*a*) Five specimens shall be tested in the condition in which they are received.

(*b*) The speed of the head of the testing machine shall be such that the load can be accurately weighed but shall not exceed 0.050 in. (1.27 mm.) per minute with the machine running idle. All tests shall be made at normal room temperature of about 20° C. (68° F.). Measurements may be taken at intervals during the test to show the elongation of the specimen when required for elastic materials.

Report.

5. The report shall include the following:

(*a*) The breaking load of each specimen in pounds or kilograms;

(*b*) The thickness of each specimen in inches or millimeters as measured by a micrometer at the center of the specimen, that is, the point of minimum section;

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials. In using these methods, reference should always be made to any pending revisions affecting the methods.

For high-frequency tests, see the Tentative Methods of Testing Electrical Insulating Materials for Power Factor and Dielectric Constant at Frequencies of 100 to 1500 Kilocycles (D 150 – 27 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); and the Tentative Method of Testing Electrical Insulating Materials for Voltage Effects at Radio Frequencies (D 175 – 25 T) of the American Society for Testing Materials, *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part I, p. 804 (1925); see also 1927 Book of A.S.T.M. Tentative Standards, pp. 599 to 614.

(*c*) The ultimate tensile strength in pounds per square inch or in kilograms per square millimeter of each specimen, calculated from the minimum area measured at the center of the test specimen before the load is applied;

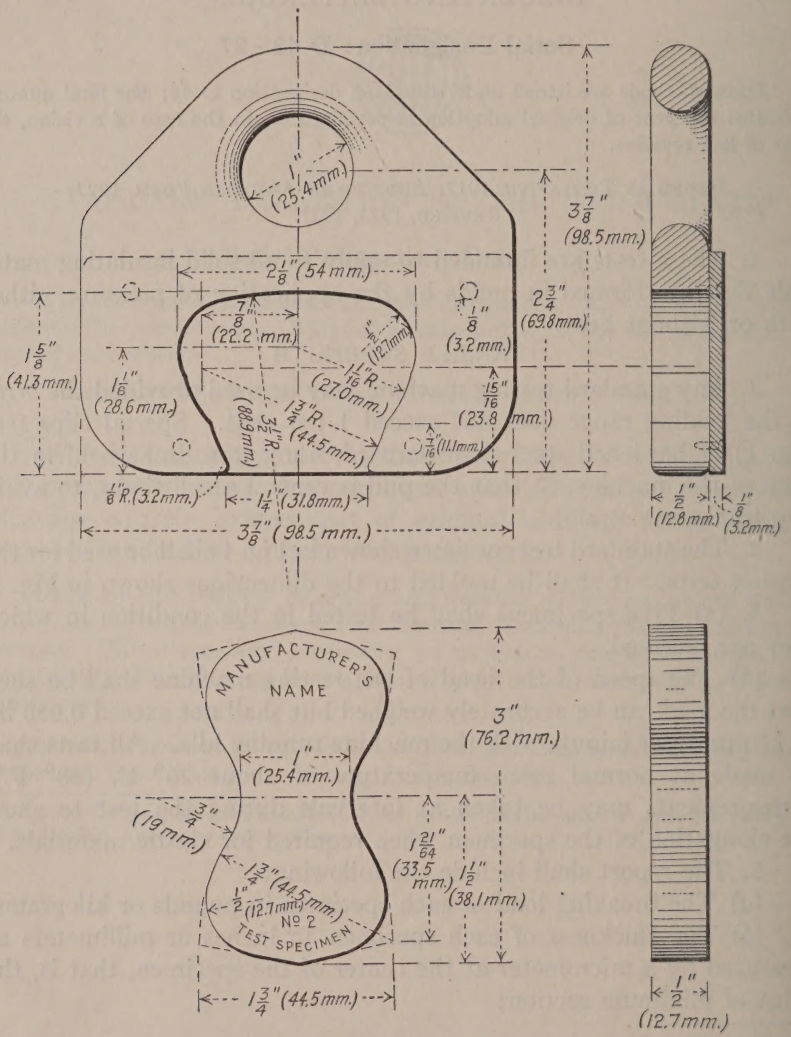

FIG. 1.—Tension Test Specimen (Specimen No. 2).

(*d*) The character of the material tested, with description of how it acts under stress;

(*e*) The speed in inches or millimeters per minute at which the jaws traveled during the test.

COMPRESSIVE STRENGTH

Apparatus.

6. Any standard testing machine may be used provided it is accurate to within 1 per cent of the lowest load for which it is used. One end of the specimen shall bear upon an accurately centered spherical bearing block, located whenever practicable at the top, and the metal bearing plates shall be directly in contact with the ends of the test specimen. The pressure heads used for standard compression tests of cement (see Tentative Specifications and Tests for Compressive Strength of Portland-Cement Mortars (Serial Designation: C 9 – 16 T) of the American Society for Testing Materials)[1] are satisfactory for this test.

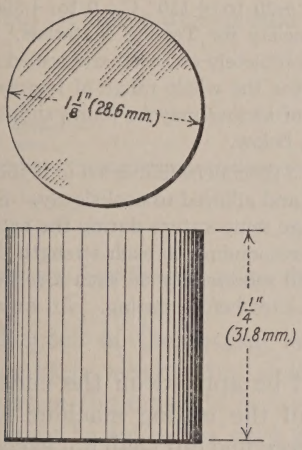

FIG. 2.—Compression Test Specimen (Specimen No. 3).

Specimen.

7. The test specimen shall be molded in the form of a cylinder having a height of 1.25 in. (31.8 mm.) and a diameter of 1.125 in. (28.6 mm.) (Fig. 2). The ends shall be flat and perpendicular to the axis, being ground to this condition if necessary.

Method.

8. (*a*) Five specimens shall be crushed in the condition in which they are received.

Additional tests shall be made at elevated temperatures, the actual temperatures selected depending upon the use that is to be made of the material. Five specimens shall be tested at each temperature selected. The specimen shall be kept at that temperature

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. XX, Part I, p. 599 (1920); also 1927 Book of A.S.T.M. Tentative Standards, p. 189.

a sufficient length of time to become of uniform temperature throughout and shall be maintained at that temperature during the test.

NOTE 1.—A convenient method of making this test is to submerge the specimens in a suitably lagged, electrically-heated oil bath of about two gallons capacity. The specimens should be placed in the bath at least 30 minutes before testing and the temperature maintained approximately constant at the desired testing temperature until the completion of that test. A pale mineral oil having a viscosity of 100 seconds ±10 seconds at 38° C. (100° F.) Saybolt is satisfactory for this purpose. The specimens should not be placed in the oil an excessive time in advance of testing. The oil bath should be well stirred to insure uniform temperature throughout. The temperature should be determined by means of a thermocouple sealed in a similar specimen in the bath; or by the use of an A.S.T.M. partial-immersion thermometer conforming to the Standard Specifications for A.S.T.M. Partial-Immersion Thermometer for General Use, −20 to +150° C., 0 to +300° F. (Serial Designation: D 182) of the American Society for Testing Materials,[1] so inserted in a hole in a specimen that the bulb is completely enclosed and sealed in.

If tests are required over the whole range of temperature rather than at particular points, the method of testing may be found simpler and quicker if modified in accordance with Note 2, below.

NOTE 2.—The oil with 5 specimens immersed in it should be heated to the highest temperature to be used and allowed to cool slowly. Specimens should be tested in succession and the average temperature during the actual time of testing of each specimen taken as that corresponding to each strength determination. Specimens should be added to the bath successively on such a schedule that each will remain in the bath at least 30 minutes before testing. An average curve shall be drawn through the plotted data.

(*b*) The load shall be applied on the ends of the specimen and the cross-head speed of the testing machine shall be such that the load can be accurately weighed but shall not exceed 0.050 in. (1.27 mm.) per minute when the machine is running idle. For the best results use the slowest possible speed.

Report. 9. The report shall include the following:

(*a*) The dimensions of each specimen in inches or in millimeters;

(*b*) The load on each specimen in pounds or kilograms at the first sign of failure;

(*c*) The ultimate compressive strength in pounds per square inch or kilograms per square millimeter, of each specimen, calculated from the measured area of each specimen before the load is applied;

(*d*) The general character of the material tested, with description of how it acts under the applied load;

(*e*) The speed in inches or millimeters per minute at which the head traveled during the test. The time required for testing each specimen should be recorded.

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[1] See p. 911.

TRANSVERSE STRENGTH

10. Any standard testing machine may be used provided it is accurate to within 1 per cent for the lowest load for which it is used. The distance between points of support shall be 4 in. (102 mm.). The supports shall have the contact edges rounded to a radius of $\frac{1}{8}$ in. (3.18 mm.). The load shall be applied midway between the supports by a pressure piece, the bearing edge of which shall be rounded to a radius of $\frac{1}{8}$ in. (3.18 mm.). **Apparatus.**

11. The test specimen shall be molded to the dimensions shown in Fig. 3. **Specimen.**

12. (*a*) Five specimens shall be tested in the condition in which they are received. **Method.**

Additional tests shall be made at elevated temperatures as outlined in Section 8.

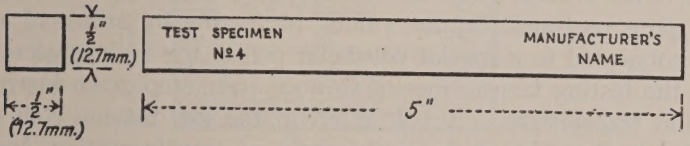

FIG. 3.—Transverse Test Specimen (Specimen No. 4).

(*b*) The cross-head speed of the testing machine shall be such that the load can be accurately weighed but shall not exceed 0.050 in. (1.27 mm.) per minute when the machine is running idle. Measurements of the deflection may be taken for very elastic materials.

13. The report shall include the following: **Report.**

(*a*) The thickness and width of each specimen as measured by a micrometer in inches or millimeters;

(*b*) The load on each specimen in pounds or in kilograms at the first sign of failure;

(*c*) The maximum fiber stress in pounds per square inch or in kilograms per square millimeter calculated from the formula:

$$S = \frac{3\,Pl}{2\,bd^2}$$

in which S = maximum fiber stress, P = load applied, l = distance between the supports, b = width of specimen, and d = depth of specimen;

(*d*) The rate at which the load was applied;

(*e*) The maximum deflection at the center in inches or in millimeters.

DIELECTRIC STRENGTH AT COMMERCIAL POWER FREQUENCIES

Apparatus. 14. (*a*) Any well-designed high-tension transformer connected to an alternating current supply having as nearly a true sine wave as possible, may be used. The transformer and the source of supply of energy shall be not less than 2 kva. for voltages of 50,000 volts or less, and not less than 5 kva. for voltages above 50,000 volts. The frequency shall not exceed 100 cycles per second.

(*b*) Regulation shall be so controlled that the high tension testing voltage taken from the secondary of the testing transformer can be raised gradually from any point and in no case more than 500 volts at a step. The control may be made by generator field regulation, with an induction regulator, or with a variable ratio auto transformer. Any method of regulating the voltage is satisfactory which does not distort the wave more than 10 per cent from a sinusoidal shape.

(*c*) The voltage may be measured by any approved method which gives root-mean-square values, preferably by means of a voltmeter connected to a special voltmeter coil in the high tension winding of the testing transformer or to a separate step-down instrument potential transformer. A voltmeter on the low tension side of the transformer is satisfactory, if the ratio of transformation does not change under any test condition. An electrostatic voltmeter properly calibrated in the high tension circuit is also satisfactory. A spark gap may be used to check the readings at very high potentials.

(*d*) Some protection is desirable in the high tension circuit of testing transformers where the potential is 25,000 volts or over, to prevent dangerous surges and limit the current when the test specimen is punctured. It is, however, desirable to have as much energy available as possible when puncture occurs. If impedance in the form of choke coils be used in series with the high tension terminals, it should not be greater than that which will limit the high tension current to double the normal rated current of the testing transformer.

When a spark gap is used, a non-inductive resistance of about one ohm per volt may be inserted in series with one terminal of the spark gap, to damp high frequency oscillations at the time of breakdown and limit the current flow. This resistance shall be as near the gap as possible. If the test is made with one side grounded, this resistance shall be on the ungrounded side of the circuit, and if neither side is grounded the resistance shall be inserted one-half on each side of the spark gap. Water tube resistors are preferable to carbon for this purpose, as carbon resistance may be materially decreased by the passage of current.

(*e*) The apparatus used and the method of measuring the voltage shall meet the requirements of the standardization rules of the American Institute of Electrical Engineers.

15. The test specimen shall be molded to the dimensions shown **Specimen.**

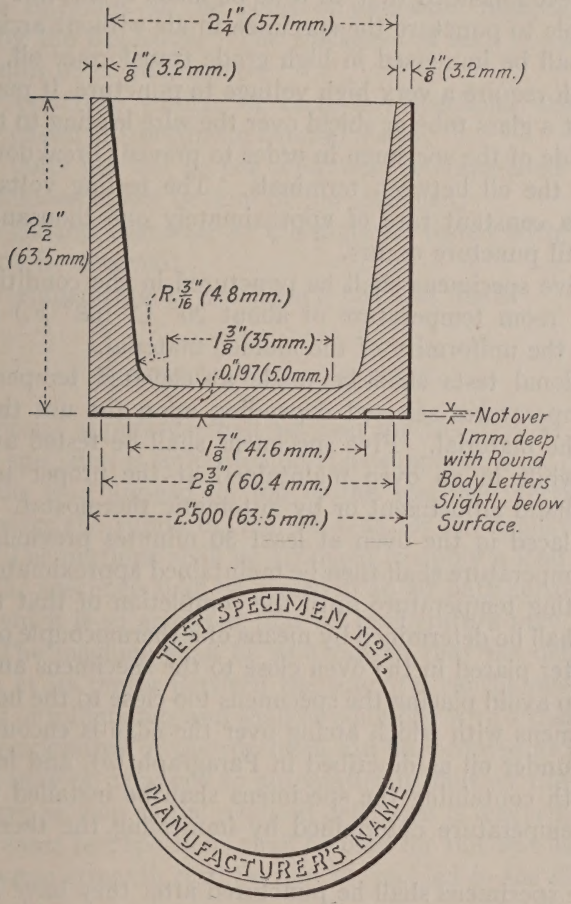

FIG. 4.—Dielectric Test Specimen (Specimen No. 1).

in Fig. 4. If the material cannot be molded to the full height shown, the height may be reduced to 1¼ in. (31.8 mm.).

For materials having a puncture value higher than 300 volts per mil, the thickness of the bottom of the specimen may be reduced to 0.098 in. (2.5 mm.). It should be noted, however, that the apparent

dielectric strength in volts per mil may be increased as much as 50 per cent when the thickness of the bottom of the specimen is so reduced.

Method. 16. (*a*) Voltage shall be applied to the test specimen by floating the specimen on mercury and placing a pool of mercury about $\frac{1}{8}$ in. (3 mm.) deep inside the specimen.

It is recommended that all tests be made in air, but whenever it is impossible to puncture the specimen in air without arcing over the edge, it shall be immersed in high grade transformer oil. On specimens which require a very high voltage to puncture, it may be necessary to put a glass tube or shield over the wire leading to the mercury on the inside of the specimen in order to prevent breakdown over the surface of the oil between terminals. The testing voltage shall be raised at a constant rate of approximately one thousand volts per second until puncture occurs.

(*b*) Five specimens shall be punctured in the condition received at normal room temperature of about 20° C. (68° F.) in order to determine the uniformity of the molded material.

Additional tests shall be made at elevated temperatures, the actual temperatures selected depending upon the use that is to be made of the material. Five specimens shall be tested at each temperature while in an oven maintained at the proper temperature, either by hand adjustment or by automatic thermostat. Specimens shall be placed in the oven at least 30 minutes previous to testing and the temperature shall then be maintained approximately constant at the testing temperature until the completion of that test. Temperature shall be determined by means of a thermocouple or a mercury thermometer placed in the oven close to the specimens and care shall be taken to avoid placing the specimens too close to the heater.

Specimens with which arcing over the edge is encountered shall be tested under oil as described in Paragraph (*a*), and in such cases the oil bath containing the specimens shall be installed in the oven and the temperature determined by immersing the thermometer in the oil.

Three specimens shall be punctured after they have had the rim immersed in melted paraffin for a depth of 1 in. (25.4 mm.) and have been entirely immersed in water for 48 hours at normal room temperature of about 20° C. (68° F.). The surface of the specimen shall be wiped off with a dry cloth to remove all trace of excessive surface moisture and the puncture test made immediately.

(*c*) The results from specimens where puncture takes place up on the side of the specimen instead of through the bottom shall be discarded. Experience shows that very plastic materials which flow

easily in the mold always puncture through the bottom, while materials which do not mold readily will often puncture through the side walls of the specimen at some distance up from the bottom.

17. The report shall include the following: Report.

(*a*) The thickness of the bottom of each specimen measured with a micrometer in the direction perpendicular to the bottom surface, and also the thickness at the point of puncture, regardless of the path taken by the discharge. The thickness of each specimen shall be given in mils or in millimeters;

(*b*) The puncture voltage of each test specimen expressed in terms of the effective (root-mean-square) value;

(*c*) The volts per mil or per millimeter as calculated from the measured thickness of the bottom of the specimen;

(*d*) The general character of the material tested with regard to leakage, if any is observed;

(*e*) The temperature at which each test was made.

DISTORTION UNDER HEAT

18. A special apparatus shall be used for this test as shown in Fig. 5. The specimen is supported on steel supports, 4 in. (102 mm.) apart with the load applied on top of the specimen vertically and midway between the supports, the same as for the transverse test, Section 10. The machine shall be arranged to apply a load of 5.5 lb. (2.5 kg.). The specimen shall be placed in an air bath surrounded by an oil bath which is so arranged that its temperature may be raised gradually. The machine shall be so arranged that the deflection of the specimen at its center between the supports can be measured on a scale in mils or millimeters and shall be equipped with a thermometer so that the temperature of the specimen can be recorded at any time. The machine may be arranged to automatically shut off the heat and sound an alarm as soon as the required deflection is reached. Apparatus.

19. The same test specimen shall be used for this test as required for the transverse strength test, Section 11, molded to the dimensions shown in Fig. 3. Specimen.

20. Three test specimens shall be tested in the condition in which they are received, starting at normal room temperature of about 20° C. (68° F.) and increasing the temperature gradually at a rate not exceeding approximately 1° C. every two minutes. Method.

The distortion point shall be considered the temperature at which the specimen has deflected 10 mils (0.254 mm.) at the center between the supports.

Report.

21. The report shall include the following:

(*a*) The breadth and depth of each specimen measured at the center with a micrometer in inches or in millimeters;

(*b*) The distortion point in degrees Centigrade or in degrees Fahrenheit;

(*c*) The length of time in minutes required for each specimen to deflect 10 mils (0.254 mm.);

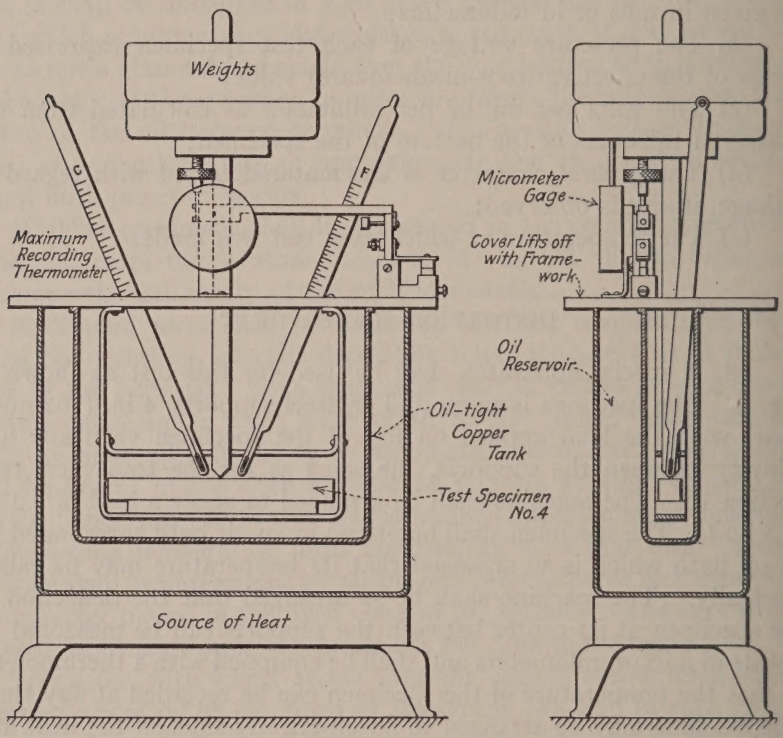

FIG. 5.—Machine for Temperature Tests.

(*d*) Any peculiar characteristics of the material as noted either during the test or after the specimen is removed from the machine.

WATER ABSORPTION

Apparatus.

22. Any good chemical balance, a beaker of water at normal room temperature of about 20° C. (68° F.), and an oven of any standard make capable of maintaining a uniform temperature of 100° C. (212° F.) with a variation allowed of ±5° C.

23. The test specimen No. 3 shown in Fig. 2 shall be used for this test. **Specimen.**

24. Three specimens shall be tested. Each of the three shall be weighed separately in the as-received condition. If the material softens readily at moderate temperatures, the specimen may be placed in a desiccator for 24 hours, or in an oven at a temperature of 50° C. (122° F.), permissible variation ±5° C., for 24 hours. For materials which do not soften readily, the specimen shall be placed in an oven heated to 100° C. (212° F.), permissible variation ±5° C., for 24 hours. After drying, the pieces shall be cooled in a desiccator and weighed again at normal room temperature. The specimens shall be placed in water, wholly immersed, for 48 hours at normal room temperature. They shall then be removed from the water at the end of 48 hours, all surface water wiped off with a dry cloth, and the specimens weighed immediately. **Method.**

25. The report shall include the following: **Report.**

(*a*) The original weight of each specimen;

(*b*) The dry weight of each specimen;

(*c*) The weight of each specimen after immersion for 48 hours;

(*d*) The percentage of water contained in each test specimen as received, and the percentage of moisture absorbed during the 48 hours, taking the dry weight as 100 per cent.

All weights shall be given in grams.

STANDARD METHODS OF TESTING ELECTRICAL INSULATING OILS[1]

Serial Designation: D 117 – 27

These methods are issued under the fixed designation D 117; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED, 1924; REVISED, 1927.

Scope.

1. These tests are intended for oil used in transformers, oil circuit breakers and other electrical apparatus as an insulating or cooling medium, or both.

SAMPLING

Sampling.

2. Some of the tests, notably dielectric strength and sludge tests,[2] of this class of material are very greatly affected by the most minute traces of certain impurities. Moisture, for example, greatly affects the dielectric test. It is exceedingly important that the samples be handled with the utmost care to avoid contamination. Therefore the following procedure and precautions shall be observed in obtaining and transporting samples of the oil to be tested.

Apparatus for Sampling.

3. (*a*) *Containers for Samples.*—Tin cans, or clear glass cork-stoppered bottles in cartons may be used. Containers shall be chemically clean and dry.

NOTE.—The glass bottle offers the advantage that it may be examined to see that it is clean. It also allows visual inspection of the oil before testing, particularly as regards free water and solid impurities. However, if samples are placed in glass bottles, they shall be kept in the dark until the portions are removed for testing for color and sludge forming characteristics, as light produces changes in these properties. Rubber stoppers should never be used with the glass containers. Only good quality cork should be used with a new cork for each sample. Contact of the oil with the cork may be prevented by means of wrapping around the cork before forcing it into the mouth of the bottle some pure thin tin foil or a similar material not attacked by oil. The tin can fitted with a screw cap protected with a non-absorbent gasket will endure harder usage. Tin cans should be soldered on the exterior surfaces.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

[2] The great importance of a sludging test is recognized but none is included in this Standard at this time because none of the methods in general use are considered sufficiently satisfactory by Committee D-9 to warrant consideration for standardization purposes. Certain new methods are being actively investigated. See reports of Committee D-9 for 1922, 1923, 1924 and 1925.

(*b*) *Thiefs.*—When sampling a shipment of oil, enough thiefs should be provided to insure drainage of each before it is used a second time. For obtaining only a few samples, two or three thiefs are enough, but for sampling a car lot of drummed oil, six or more are desirable.

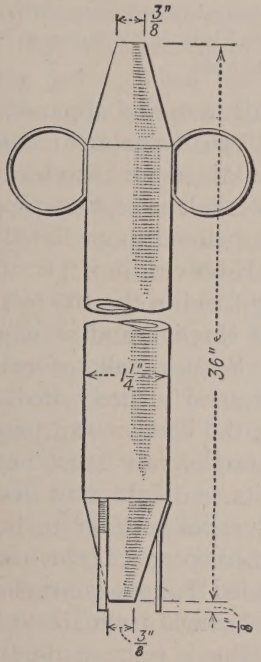

FIG. 1.—Drum Thief.

NOTE.—A convenient and simple thief (see Fig. 1) for use with 50-gal. drum containers may be made of tin as follows: Length 36 in. and diameter 1¼ in., with cone-shaped caps over the ends and having openings at the ends ⅜ in. in diameter. Three legs equally spaced around the thief at the bottom, long enough to hold the opening ⅛ in. from the bottom of container being sampled, aids in securing a good representative sample. Two rings soldered to opposite sides of the tube at the upper end will be found convenient for holding the thief by slipping two fingers through them, leaving the thumb free to close the opening. For tank cars, a thief employing a trap at the bottom may be used.

Cleaning of Sampling Apparatus.

4. All thiefs and sample receptacles shall be thoroughly cleaned before using, special care being taken that no lint or other fibrous material remains in or on them.

NOTE.—It is suggested that the thiefs be cleaned by rinsing with oil-free gasoline and dried in the hot cabinet (see Section 5) before using. The best procedure is to rinse them after the sampling has been finished and then place them in the hot

cabinet. They will in this way be ready for use without further cleaning when the next shipment is to be sampled.

All sample containers before using should be rinsed with oil-free gasoline and dried. They should then be washed with strong soapsuds, rinsed thoroughly with water and then dried in an oven at 105 to 110° C. A similar washing will be sufficient for re-use of the containers for samples of new oil, but those for used oil should be washed with both gasoline and benzol before using soapsuds. Containers which are not chemically clean and dry are likely to contaminate the sample of oil and cause misleading results in some of the tests.

Storage of Sampling Apparatus.

5. When not in use all sampling apparatus should be kept in a hot, dry, dust-free cabinet or compartment at a temperature not less than 38° C. (100° F). Containers for samples, after washing and drying as indicated in Section 4, shall be kept unstoppered while in the oven or hot cabinet but shall be closed immediately after they are taken out of the hot cabinet. However, it is permissible to prepare containers in advance, leaving them in the 105 to 110° C. oven until dry, then removing, corking or capping and sealing by dipping the neck into melted paraffin wax. Bottles shall, before dipping, have cork and neck wrapped with paper, and securely tied. If this procedure is followed, before removing the cork or cap preparatory to filling the bottle or can with the sample, care must be taken to remove the paraffin so as to prevent its contaminating the sample. Thiefs shall be kept at all times in a vertical position in the hot cabinet in a rack having a suitable drainage receptacle at the base.

Procedure for New Oils.

6. (*a*) It is recommended that one quart of oil be taken as a sample if complete physical and chemical tests are to be made; at least one pint if the dielectric test alone is to be made.

(*b*) Drums or tank cars, etc., of oil shall not be sampled until the oil is at least as warm as the surrounding air. Electrical insulating oil is not hygroscopic but cold oil may condense enough moisture on the surface from a humid atmosphere to seriously affect its insulating properties.

(*c*) Samples of oil shall be taken only after the oil has remained undisturbed and has settled for at least 8 hours. Samples must, of course, never be taken in the rain and precautions must be taken during sampling to prevent contamination by wind-blown dust, etc.

(*d*) Drums and barrels of oil to be sampled should be lined up, preferably on their sides, and numbered, bungs up. The bungs shall be unsealed and removed and laid with the oily side up beside the bung holes. The unstoppered sample receptacle may be placed on the opposite side of the bung holes. The top hole of the thief shall be closed with the thumb, the thief thrust to the lowest point or recess of the container and the thumb removed. When the thief is

filled the thumb shall be replaced, the thief quickly withdrawn and the contents allowed to flow into the sample receptacle. The lower holes shall not be closed with the fingers of the other hand. The free hand shall be used to guide the stream of oil by touching the thief only as necessary. The oil shall not be allowed to flow over the hand or fingers before it flows into the sample receptacle nor shall any excess be allowed to flow over the drum and back into the drum through the open bung hole. When the sample receptacle is filled it shall be closed quickly and the bung replaced in the container and tightened. The sample receptacle shall be taken to the testing laboratory as quickly as feasible.

(*e*) Tank cars of oil shall be sampled with the thief, introducing it through the manhole on top of the car, the cover of which shall be removed carefully so as not to introduce any dirt into the oil. The sample shall be taken as near as possible to the bottom of the tank car.

Procedure for Oil in Service.

7. (*a*) When taking samples of oil from transformers, oil circuit breakers and other apparatus where a thief cannot be used, the cautions outlined in Section 6 shall be observed so far as practicable. In addition, care shall be taken to procure a sample which fairly represents the oil at the bottom of the tank. Transformer and oil circuit-breaker tanks, etc., are usually provided with drain pipes or sampling cocks from which the sample can be allowed to flow into the sample container. Sufficient oil should be allowed to flow to waste before the test sample is taken to insure that the sample will not be that portion which was stored in the drain pipe. For this reason the valve and the drain pipe should be sufficiently small to be emptied with convenience and yet sufficiently large to give an even flow of oil and avoid clogging by sediment. A $\frac{1}{4}$-in. pipe and valve are recommended. This, of course, may be separate from the drainage pipe and valve or may be connected to the drainage valve by means of a suitable reducer.

VISCOSITY

Viscosity.

8. The viscosity test shall be made in accordance with the Standard Methods of Test for Viscosity of Petroleum Products and Lubricants (Serial Designation: D 88) of the American Society for Testing Materials[1] and unless otherwise directed shall be taken at a temperature of 37.8° C. (100° F.).

FLASH POINT

Flash Point.

9. The flash point shall be determined in accordance with the Standard Method of Test for Flash and Fire Points by Means of

[1] See p. 427.

Open Cup (Serial Designation: D 92) of the American Society for Testing Materials.[1]

POUR POINT

Pour Point.

10. The pour point shall be determined in accordance with the Tentative Method of Test for Cloud and Pour Points of Petroleum Products (Serial Designation: D 97 – 27 T) of the American Society for Testing Materials.[2]

NEUTRALIZATION NUMBER

Neutralization Number.

11. The neutralization number shall be determined in accordance with the Tentative Method of Test for Neutralization Number of Petroleum Products and Lubricants (Serial Designation: D 188 – 27 T) of the American Society for Testing Materials,[3] using method A, Section 4.

MINERAL ACIDS (CHLORIDES AND SULFATES)

Mineral Acids.

12. The presence of mineral acids (Cl or SO_3) shall be determined by thoroughly shaking in a separatory funnel, 100 cc. of the oil with 50 to 100 cc. of hot (90 to 100° C.) distilled water and then separating and drawing off the water layer and testing it qualitatively with the usual procedures for the presence of chlorides or sulfates.

FREE AND CORROSIVE SULFUR

Apparatus.

14. (*a*) The apparatus for this determination should consist of the following:

(*a*) Electrolytic sheet copper: approximate dimensions.—length, 3 in. (7.62 cm.); width, 0.5 in. (1.27 cm.); thickness, 0.031 in. (0.079 cm.).

(*b*) Glass tube: acid-resistant glass, 125 cc. capacity, fitted with a slotted cork (a test tube of suitable size is satisfactory).

(*c*) Suitable bath in which a temperature of from 95 to 110° C. can be maintained.

Reagents.

15. *Reagents.*—The following reagents will be required:

(*a*) Fuming nitric acid, c. p.;

(*b*) Hydrochloric acid, c. p. concentrated;

(*c*) Barium chloride, 10-per-cent solution.

Procedure.

16. The copper strip shall be mechanically polished. It shall be thoroughly washed with distilled water to remove the polishing compounds and dried between filter paper. The polished copper strip

[1] See p. 404.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M Tentative Standards, p. 390.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I (1927); also 1927 Book of A.S.T.M. Tentative Standards, p. 418.

shall be placed in the glass tube and 100 cc. of oil added. The strip shall be completely immersed. The tube shall be closed with a vented cork and the oil heated in the bath for not less than five hours, maintaining on the oil sample a temperature between 95 and 110° C.

If free sulfur or corrosive sulfur compounds are present, the copper strip will be discolored. This discoloration (a dark characteristic film) is not definite proof of the presence of free sulfur or corrosive-sulfur compounds. It shall be tested as follows:

Remove the oil from the copper strip by rinsing with petroleum ether. After the ether has evaporated, completely dissolve the discoloring film with a few drops of fuming nitric acid, catching the solution in a 50-cc. beaker. In this manner a minimum of copper is removed. Wash the strip with a minimum of distilled water, catching the washings in the beaker. To the combined solution, add about 5 cc. of concentrated hydrochloric acid. Boil the solution to drive off free nitric acid. If necessary, dilute with a small amount of distilled water, and then to the boiling solution add 5 cc. of the 10-per-cent solution of barium chloride. A white precipitate proves the presence of sulfur. Minute traces of sulfur are not precipitated immediately. If no precipitate forms in six hours, the temperature of the solution having been maintained at about 95° C., free sulfur or corrosive-sulfur compounds shall be considered absent.

NOTE.—All reagents shall be sulfur-free. A blank should be run with each batch of determinations, following the above procedure, but using water in equal volume instead of oil. With necessary precautions the volume of solution for proving the presence or absence of sulfur should not exceed 20 to 30 cc. throughout the manipulation.

DIELECTRIC STRENGTH

Apparatus.

17. (*a*) The apparatus shall be as described in Section 14 of the Standard Methods of Testing Molded Insulating Materials (Serial Designation: D 48) of the American Society for Testing Materials.[1] The test cup for holding the sample of oil shall be made of a material having a suitable dielectric strength. It must be insoluble in and unattacked by mineral oil and gasoline and non-absorbent as far as moisture, mineral oil and gasoline are concerned. The electrodes in the test cup between which the sample is tested shall be circular disks of polished brass or copper, one inch in diameter and having square edges. The electrodes shall be mounted in the test cup having their axes horizontal and coincident, with a gap of 0.1 in. between their adjacent faces, and with top of electrodes about 33 mm. (1¼ in.) below the top of the cup.

[1] See p. 785.

Procedure.

18. (*a*) The electrodes and the test cup shall be wiped clean with dry calendered tissue paper or with a clean dry chamois skin and thoroughly rinsed with oil-free dry gasoline until they are entirely free from fibers.

(*b*) The spacing of electrodes shall be checked with a standard round gage having a diameter of 0.100 in. and the electrodes then locked in position. Care shall be taken not to touch the electrodes with the gage or in any other manner after cleaning the electrodes and cup, so as to avoid any possible contamination.

(*c*) The test cup shall be filled with dry gasoline, and voltage applied with uniform increase at the rate of approximately 3 kv. (rms.) per second until breakdown occurs. If the dielectric strength is not less than 25 kv., the cup shall be considered in suitable condition for testing the oil. If a lower test value is obtained the cup shall be cleaned with gasoline and the test repeated.

NOTE.—Evaporation of gasoline from the electrodes may chill them sufficiently to cause moisture to condense on their surface. For this reason, after the final rinsing with gasoline, the test cup should be immediately filled with the oil which is being tested and the test proceeded with at once, or the electrodes should be thoroughly dried.

(*d*) The temperature of the oil when tested shall be the same as that of the room which should be between 20 and 30° C. (68 and 86° F.). Testing at lower temperatures is likely to give variable results which may be misleading.

(*e*) The sample in the container shall be shaken so as to thoroughly mix the oil before filling the test cup. This is even more important with used than with new oil as the impurities may settle to the bottom and the test may be misleading.

(*f*) The cup shall be filled with oil to a height of not less than 20 mm. (0.787 in.) above the top of the electrodes.

(*g*) The oil shall be gently agitated by rocking the cup and then the oil shall be allowed to stand in the cup for three minutes before the first and one minute before each succeeding puncture. This will allow air bubbles to escape.

(*h*) Voltage shall be applied and increased uniformly at a rate of approximately 3 kv. (rms.) per second until breakdown occurs as indicated by a continuous discharge across the gap. (Occasional momentary discharges which do not result in a permanent arc may occur; these should be disregarded.)

(*i*) Provision shall be made for opening the circuit as promptly as possible after breakdown has occurred in order to prevent unnecessary carbonization of the oil. After each puncture, the testing vessel

shall be jarred to loosen particles of carbon adhering to the electrodes and the oil gently agitated but not with sufficient violence to introduce air bubbles.

(*j*) Five breakdowns shall be made on each filling after which the vessel shall be emptied and refilled with fresh oil from the original sample. The test shall be continued until the averaged values of at least three fillings do not differ from their mean by more than 10 per cent.

(*k*) The last filling of the cup should drain the container. If a quart sample be received, allowance shall be made in withdrawing portions so that just enough be left for a final filling of the cup with the drainings from the container.

19. The report shall include the following: Report.

(*a*) The volts (rms. value) at each puncture, the average voltage for each of the three or more fillings and the grand average;

(*b*) The report shall be stated as kv., 1.0 in. disk 0.1 in. gap;

(*c*) The approximate temperature of the oil at the time of the test.

NOTE.—A precision of about 3 per cent may reasonably be expected in 15 tests distributed among three consistent fillings taken in succession. But if the length of the gap is readjusted and possibilities of contamination exist, the precision may be only 6 or 7 per cent. Differences as great as 10 or 12 per cent may occur between different laboratories even where the work is carefully done. (See Appendix I to Report of Committee D-9 of the American Society for Testing Materials in 1921, *Proceedings*, Am. Soc. Testing Mats., Vol. 21, p. 397 (1921).)

STANDARD SPECIFICATIONS
FOR
COTTON RUBBER-LINED FIRE HOSE FOR PRIVATE FIRE DEPARTMENT USE.[1]

Serial Designation: D 14 – 23

These specifications are issued under the fixed designation D 14; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1915; REISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1923.

Scope.

1. These specifications apply to cotton rubber-lined hose for private use for fire protection purposes. They cover the following sizes: 1½-in. single and double-jacketed; 2-in. single and double jacketed; 2½-in. single, double and triple-jacketed; 3-in. double and triple-jacketed; 3½-in. double and triple-jacketed.

MANUFACTURE

Cotton Fabric.

2. (*a*) The jacket or jackets shall be well, evenly and firmly made from good cotton, as free from unsightly defects, dirt, knots, lumps and irregularities of twist as is consistent with good manufacturing practice.

(*b*) The cotton jackets shall be seamless and have the fillers woven around the hose throughout its length and the warps interwoven with and covering the fillers. Where more than one jacket is prescribed, they may be separate or interwoven.

Rubber Lining.

3. (*a*) The lining shall be made a properly vulcanized rubber compound which will comply with all of the tests specified in Section 8. It shall consist of not less than three calendered sheets in one solid body and shall be lap-jointed with the lap as small and neat as is consistent with good results.

(*b*) The lining shall be smooth and practically free from pitting and other imperfections and from corrugations.

(*c*) The lining shall be of uniform thickness and not less than the lower limit nor more than the upper limit given in item (*b*) of Table I, exclusive of the backing.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

(*d*) The backing, if used, shall not exceed 0.028 in. in thickness. It need not be of the same composition as the rubber lining, but the adhesion between the rubber lining and the cotton jacket shall comply with the test specified in Section 9.

4. The hose shall be flexible and easily coiled. **Flexibility.**

5. The character of the workmanship shall be such as is incident to good manufacturing practice. **Workmanship.**

TABLE I

Property.	Size and Number of Jackets.										
	1½-in		2-in.		2½-in.			3-in.		3½-in.	
	Single.	Double.	Single.	Double.	Single.	Double.	Triple.	Double.	Triple.	Double.	Triple.
(*a*) Prescribed internal diameter in.	1½	1½	2	2	2½	2½	2½	3	3	3½	3½
(*b*) Thicknes,srubber lining, in.	0.049 to 0.065	0.049 to 0.065	0.049 to 0.065	0.049 to 0.065	0.058 to 0.072	0.058 to 0.072	0.058 to 0.072	0.058 to 0.072	0.058 to 0.072	0.072 to 0.095	0.072 to 0.095
(*c*) Test pressure on every length, lb. per sq. in.	300	400	300	400	300	400	400	400	400	400	400
(*d*) Bursting test pressure (3-ft. samples) lb. per sq. in.	500	600	500	600	500	600	600	600	600	600	600
(*e*) Kink test pressure, lb. per sq in.	250	300	250	300	250	300	300	250	250	250	250
(*f*) Elongation, per cent.	13	9	13	9	13	9	9	9	9	9	9
(*g*) Twist, deg. per ft.	50	29	50	29	25	15	15	15	15	15	15
(*h*) Rise in	7	none	7	none	4	none	none	none	none	none	none
(*i*) Weight, min., lb.	21	..	29	..	40	..	..	..	..	..	..
Weight, max., lb	..	42	..	58	..	70	86	93	113	115	135
(*j*) Test pressure marking, lb. per sq. in.	300	400	300	400	300	400	400	400	400	400	400

PHYSICAL PROPERTIES AND TESTS

6. The tests necessary to determine the physical properties which are prescribed in the following sections shall be made in accordance with the Standard Methods of Testing Rubber Products (Serial Designation: D 15) of the American Society for Testing Materials.[1] **Standard Methods.**

7. A sample not less than 8 in. in length shall be taken from each lot of 500 ft. of hose or less for the tests specified in Sections 8 and 9. All test specimens shall be cut transversely from the sample. **Sampling.**

8. (*a*) The tensile strength shall be not less than 1600 lb. per sq. in. when tested one month or less from date of final vulcanization, and not less than 1200 lb. per sq. in. when tested more than one month but not over one year from date of final vulcanization. **Tests of Rubber Lining.**

(*b*) The tensile strength after subjection to a dry heat of 158° F. (70° C.) ±2° F. (1.1° C.) for a period of four successive days of 24 hours each shall be not less than 900 lb. per sq. in. This test shall be made within three months from date of final vulcanization.

[1] See p. 847.

(*c*) The elongation when rupture occurs in making the tension test (Paragraph (*a*)) shall be such that the original 2-in. gage length shall stretch to not less than 12 in., when tested one month or less from date of final vulcanization, and not less than 10 in. when tested more than one month but not over one year from date of final vulcanization.

(*d*) The permanent elongation or set following a stretch from 2 to 10 in. shall not exceed 25 per cent when tested one month or less from date of vulcanization. When tested more than one month but not over one year from date of final vulcanization, it shall not exceed 25 per cent following a stretch from 2 to 8 in.

Friction Test. 9. The adhesion between the lining and the cotton jacket shall be such that the rate of separation of the lining from the jacket shall be not greater than one inch per minute with a weight of 12 lb.

Hydrostatic Pressure Tests. 10. (*a*) Each length of hose shall be subjected to the hydrostatic pressure specified in item (*c*) of Table I for five seconds without leaking in the hose or at the couplings or breaking any threads in the jacket.

(*b*) A 3-ft. sample in every lot of 3000 ft. of hose or less shall, while lying either straight or curved to a radius of 27 in., withstand without failure the hydrostatic pressure specified in item (*d*) of Table I.

(*c*) One full length in every lot of 500 ft. of hose or less shall, while kinked, withstand without failure the hydrostatic pressure specified in item (*e*) of Table I applied for five seconds.

Elongation. 11. The elongation of the hose when tested as prescribed in Section 10 (*a*) shall not exceed the percentage of the length measured under a pressure of 10 lb. per sq. in. which is specified in item (*f*) of Table I.

Twisting. 12. When tested as prescribed in Section 10 (*a*), the hose shall not twist more than the amount given in item (*g*) of Table I. The twisting shall be in the direction which will tighten rather than loosen the couplings.

Warping and Rise. 13. When subjected to the test prescribed in Section 10 (*a*), the hose shall not rise more than the distances specified in item (*h*) of Table I, from the level of the test table or test floor or warp more than 20 in. from a straight line drawn from center to center of the couplings.

CHEMICAL PROPERTIES AND TESTS

Methods. 14. The chemical tests required by the following chemical requirements shall be made in accordance with the Standard Methods of Testing Rubber Products (Serial Designation: D 15) of the American Society for Testing Materials.[1]

[1] See p. 847.

15. Samples shall be taken both as regards number and location at the discretion of the inspector. Sampling.

16. (*a*) In the analysis of the rubber lining, the acetone extract shall not exceed 4 per cent; the alcoholic potash extract shall not exceed 1½ per cent; the chloroform extract shall not exceed 2 per cent; the ash shall be not less than 50 per cent nor more than 57 per cent; the total sulfur (excluding barytes) shall not exceed 4 per cent; and the free sulfur shall not exceed 1¼ per cent. All percentages shall be by weight of the total compound. Analysis of Rubber Lining.

(*b*) The sum total of the results of the tests of the rubber lining for acetone extract, alcoholic potash extract, chloroform extract, ash and total sulfur (exclusive of barytes) shall not exceed 67 per cent by weight of the total compound.

DIMENSIONS AND WEIGHTS

17. The hose shall have an internal diameter of not less than the diameter ordered and shall be furnished in lengths averaging 50 ft. Diameter and Length.

18. The weight of each 50-ft. length of hose complete with couplings shall be in accordance with the amount specified in item (*i*) of Table I. Weight.

COUPLINGS

19. Couplings shall be of the type ordered but unless otherwise specified, they shall conform to Sections 20 to 25, inclusive, and Section 28. Type.

20. The couplings shall be made, finished and fitted in a workmanlike manner throughout. The inside diameter throughout the couplings shall be not less than the nominal inside diameter of the hose to which they are connected. Workmanship and Diameter.

21. The couplings shall be of the expansion-ring pattern, of ample strength, and of the best form to resist the stress of expanding the binding ring in the coupling, and shall have a tail part sufficiently long to extend ⅛ in. beyond the end of the expansion ring. The ring shall be at least 1¼ in. in length. Type and Dimensions.

22. The couplings shall be made of an alloy of copper, tin and zinc (and lead if desired) of the following proportions: Chemical Composition.

Copper	not under	83	per cent	
Tin	"	" 5	"	
Zinc	"	over 7	"	
Lead	"	" 3	"	

23. The weight of complete couplings, including male and female ends, swivel, and the expansion rings, shall be not less than the values specified in Table II. Weight.

Gasket and Washer.

24. (*a*) The swivels of the couplings shall be provided with rubber gaskets of the same quality as the rubber lining, at least $\frac{3}{16}$ in. thick, and accurately fitted.

(*b*) The couplings shall have, inside each expansion ring, a rubber washer at least $\frac{3}{16}$ in. in thickness and with inside diameter not less than that of the coupling.

(*c*) The inside diameter of the gasket and the washers when in position in the couplings shall be not less than the inside diameter of the hose to which the coupling is attached.

Thread.

25. Wherever practicable and not otherwise specified, the thread shall be the "National (American) Standard Fire-Hose Coupling Screw Thread" as approved by the American Engineering Standards Committee, May, 1925.

NOTE.—This thread is the same as that originally developed by the National Fire Protection Association and known as the "National Standard Fire-Hose Thread."

TABLE II.—WEIGHT OF COUPLINGS.

NOMINAL DIAMETER OF HOSE, IN.	WEIGHT, COMPLETE, LB.		
	SINGLE JACKET.	DOUBLE JACKET.	TRIPLE JACKET.
$1\frac{1}{2}$	1.5	2.0	....
2	2.5	3.0	....
$2\frac{1}{2}$	4.0	5.0	....
3	...	8.0	10.0
$3\frac{1}{2}$	...	12.0	14.0

Attachment of Couplings.

26. The couplings shall be attached to the hose in a workmanlike manner without cutting the lining or rupturing the cotton jacket. They shall withstand the hydrostatic pressure tests described in Section 10 (*a*) without the use of tape or rubber tissue as a filler.

MARKING

Hose.

27. Beginning at a point approximately 4 ft. from the couplings, each 50-ft. length of hose shall be stenciled at two places in black indelible letters 1 in. high with the legend "A.S.T.M. Specifications," the name of the manufacturer, the month and year of manufacture, and the words "Tested at .... lb.," the pressure stated to be that given in item (*j*) of Table I.

Couplings.

28. Both male and female couplings shall be stamped with the name of the manufacturer and the month and year of manufacture in letters and figures not less than $\frac{1}{8}$ in. high.

INSPECTION AND REJECTION

29. (*a*) The manufacturer shall notify the purchaser sufficiently in advance of the completion of the hose to permit of arrangement for inspection. Inspection.

(*b*) The manufacturer shall afford the inspector representing the purchaser, without charge, all reasonable facilities to satisfy him that the hose is being furnished in accordance with these specifications. All tests and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

(*c*) The purchaser may make the tests to govern the acceptance or rejection of the hose in his own laboratory or elsewhere. Such tests, however, shall be made at the expense of the purchaser.

30. (*a*) Each length of hose that fails in the tests specified in Sections 10 (*a*), 11, 12, or 13, or any other requirement of these specifications shall be rejected. Rejection.

(*b*) In the case of failure to pass any one of the tests specified in Sections 8, 9, 10 (*b*) or 10 (*c*), the entire lot of hose represented by the sample, or samples subjected to these tests shall be rejected.

31. Samples of rejected hose shall be preserved for two weeks from the date of test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time. Rehearing.

STANDARD SPECIFICATIONS
FOR
COTTON RUBBER-LINED FIRE HOSE FOR PUBLIC FIRE DEPARTMENT USE[1]

Serial Designation: D 26 – 23

These specifications are issued under the fixed designation D 26; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1916; ADOPTED, 1918; REISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1923.

Scope.

1. These specifications apply to $2\frac{1}{2}$, 3 and $3\frac{1}{2}$-in, double-jacketed cotton, rubber-lined fire hose for use in public fire departments.

MANUFACTURE

Cotton Fabric.

2. (*a*) The two jackets shall be well, evenly and firmly made from good cotton, as free from unsightly defects, dirt, knots, lumps and irregularities of twist as is consistent with good manufacturing practice.

(*b*) Each jacket shall be seamless and have the fillers woven around the hose throughout its length and the warps interwoven with and covering the fillers. The two jackets may be separate or interwoven.

Rubber Lining.

3. (*a*) The lining shall be made of a properly vulcanized rubber compound which will comply with all the tests specified in Section 7. It shall consist of not less than three calendered sheets in one solid body and shall be lap-jointed with the lap as small and as neat as is consistent with good results.

(*b*) The lining shall be smooth and practically free from pitting and other imperfections and from corrugations.

(*c*) The lining shall be of uniform thickness and within the following limits of thickness exclusive of backing:

INTERNAL DIAMETER OF HOSE, IN.	MINIMUM THICKNESS, IN.	MAXIMUM THICKNESS, IN.
$2\frac{1}{2}$	0.058	0.072
3	0.058	0.072
$3\frac{1}{2}$	0.072	0.095

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

(*d*) The backing, if used, shall not exceed 0.028 in. in thickness. It need not be of the same composition as the rubber lining but the adhesion between the rubber lining and the cotton jacket shall comply with the test specified in Section 9.

4. The hose shall be flexible and easily coiled. **Flexibility.**

5. The character of the workmanship shall be such as is incident to good manufacturing practice. **Workmanship.**

PHYSICAL PROPERTIES AND TESTS

6. The tests necessary to determine the physical properties which are prescribed in the following sections shall be made in accordance with the Standard Methods of Testing Rubber Products (Serial Designation: D 15) of the American Society for Testing Materials.[1] **Standard Methods.**

7. A sample not less than 8 in. in length shall be taken from each lot of 500 ft. of hose or less for the tests specified in Sections 8 and 9. All test specimens shall be cut transversely from the sample. **Sampling.**

8. (*a*) The tensile strength shall be not less than 1600 lb. per sq. in. when tested one month or less from date of final vulcanization and not less than 1200 lb. per sq. in. when tested more than one month but not over one year from date of final vulcanization. **Tests of Rubber Lining.**

(*b*) The tensile strength after subjection to a dry heat of 158° F. (70° C.) ±2° F. (1.1° C.) for a period of four successive days of 24 hours each shall be not less than 900 lb. per sq. in. This test shall be made within three months from date of final vulcanization.

(*c*) The elongation when rupture occurs in making the tensile test (Paragraph (*a*)) shall be such that the original 2-in. gage length shall stretch to not less than 12 in. when tested one month or less from date of final vulcanization and not less than 10 in. when tested more than one month but not over one year from date of final vulcanization.

(*d*) The permanent elongation or set following a stretch from 2 to 10 in. shall not exceed 25 per cent when tested one month or less from date of vulcanization. When tested more than one month but not over one year from date of final vulcanization, the stretch shall be 2 to 8 in. instead of 2 to 10 in.

9. The adhesion between the lining and the cotton jackets shall be such that the rate of separation of the lining from the jackets shall be not greater than 1 in. per minute with a weight of 12 lb. **Friction Test.**

10. (*a*) Each length of hose shall be subjected to a hydrostatic pressure of 400 lb. per sq. in. for five seconds without leaking in the hose or at the couplings or breaking any threads in the jackets. **Hydrostatic Pressure Tests.**

[1] See p. 847.

(*b*) A 3-ft. sample in every lot of 3000 ft. of hose or less shall, while lying either straight or curved to a radius of 27 in., withstand without failure a hydrostatic pressure of 600 lb. per sq. in.

(*c*) One full length in every lot of 500 ft. of hose or less shall, while kinked, withstand without failure a hydrostatic pressure of 300 lb. per sq. in. for 2½-in. hose and 250 lb. per sq. in. for 3 and 3½-in. hose applied for 5 seconds.

Elongation. 11. The elongation of the hose when tested as prescribed in Section 10 (*a*) shall not exceed 9 per cent of the length, measured under a pressure of 10 lb. per sq. in.

Twisting. 12. The hose shall not twist more than 15 deg. per ft. when tested as prescribed in Section 10 (*a*). The twisting shall be in the direction which will tighten rather than loosen the couplings.

Warping and Rise. 13. When subjected to the test prescribed in Section 10 (*a*), the hose shall not rise from the level of the test table or test floor, or warp more than 20 in. from a straight line drawn from center to center of the couplings.

CHEMICAL PROPERTIES AND TESTS

Methods. 14. The chemical tests required by the following chemical requirements shall be made in accordance with the Standard Methods of Testing Rubber Products (Serial Designation: D 15) of the American Society for Testing Materials.[1]

Sampling. 15. Samples shall be taken both as regards number and location at the discretion of the inspector.

Analysis of Rubber Lining. 16. (*a*) In the analysis of the rubber lining, the acetone extract shall not exceed 4 per cent; the alcoholic potash extract shall not exceed 1½ per cent; the chloroform extract shall not exceed 2 per cent; the ash shall be not less than 50 per cent nor more than 57 per cent; the total sulfur (excluding barytes) shall not exceed 4 per cent; and the free sulfur shall not exceed 1¼ per cent. All percentages shall be by weight of the total compound.

(*b*) The sum total of the results of the tests of the rubber lining for acetone extract, alcoholic potash extract, chloroform extract, ash and total sulfur (exclusive of barytes) shall not exceed 67 per cent by weight of the total compound.

DIMENSIONS AND WEIGHTS

Diameter and Length. 17. The hoses hall have an internal diameter of not lesst han 2½, 3 or 3½ in. as ordered and shall be furnished in lengths averaging 50 ft.

[1] See p. 847.

18. The weight of each 50-ft. length of hose complete with couplings shall not exceed the following limits: **Weight.**

INTERNAL DIAMETER OF HOSE, IN.	MAXIMUM WEIGHT PER 50-FT. LENGTH, LB.
2½	70
3	93
3½	115

COUPLINGS

19. Couplings shall be as ordered but unless otherwise specified they shall conform to Sections 20 to 25, inclusive, and Section 28. **Type.**

20. The couplings shall be made, finished and fitted in a workmanlike manner throughout. The diameter through couplings shall be according to the size of the hose: namely 2½, 3 or 3½ in. **Workmanship and Diameter.**

21. The couplings shall be of the expansion-ring pattern, of ample strength, and of the best form to resist the stress of expanding the binding ring in the coupling. and shall have a tail part sufficiently long to extend ⅛ in. beyond the end of the expansion ring. They shall be fitted with expansion rings of the following lengths: **Type and Dimensions.**

INTERNAL DIAMETER OF HOSE, IN.	LENGTH OF EXPANSION RINGS, IN.
2½	1½
3	1¾
3½	2½

22. The couplings shall be made of an alloy of copper, tin and zinc (and lead if desired) of the following proportions: **Chemical Composition.**

Copper	not under	83	per cent	
Tin	"	"	5	"
Zinc	"	over	7	"
Lead	"	"	3	"

23. The weight of couplings, including both male and female and expansion rings, shall conform to the following minimum requirements: **Weight.**

INTERNAL DIAMETER OF HOSE, IN.	MINIMUM WEIGHT PER SET OF COUPLINGS, LB.
2½	5¾
3	8
3½	12½

24. (*a*) The couplings shall be provided with rubber gaskets of the same quality as the rubber lining, and accurately fitted. **Gasket and Washer.**

(*b*) The couplings shall have, inside each expansion ring, a rubber

washer with inside diameter not less than that of the coupling and thickness not less than the following:

INTERNAL DIAMETER OF HOSE, IN.	THICKNESS OF WASHER, IN.
$2\frac{1}{2}$	$\frac{3}{16}$
3	$\frac{1}{4}$
$3\frac{1}{2}$	$\frac{1}{4}$

Thread.

25. The threads of hose couplings shall be interchangeable with those in use in the city or town where the hose will be used. Wherever practicable the thread shall be the "National (American) Standard Fire-Hose Coupling Screw Thread" as approved by the American Engineering Standards Committee, May, 1925.

NOTE.—This thread is the same as that originally developed by the National Fire Protection Association and known as the "National Standard Fire-Hose Thread."

Attachment of Couplings.

26. The couplings shall be attached to the hose in a workmanlike manner without cutting the lining or rupturing the cotton jacket. They shall withstand the hydrostatic pressure tests described in Section 10 (*a*) without the use of tape or rubber tissue as a filler.

NOTE

It is recommended that couplings be ordered to conform to the Standard Fire-Hose Coupling of the National Fire Protection Association as described in Sections 20 to 25 and be provided with the National (American) Fire-Hose Coupling Screw Thread in accordance with Section 25.

SIZE OF COUPLINGS, IN.	NUMBER OF THREADS PER IN.	OUTSIDE DIAMETER OF MALE THREADS, IN.
$2\frac{1}{2}$	$7\frac{1}{2}$	$3\frac{1}{16}$
3	6	$3\frac{5}{8}$
$3\frac{1}{2}$	6	$4\frac{1}{4}$

In many cases where the number of threads and the outside diameters of the male ends do not differ greatly from the Standard, it is possible to render both male and female couplings adaptable for interchange with the Standard by the use of an adjustable tap for the female end or an adjustable die for the male end of the coupling. This tap or die should have the same number of threads per inch as the coupling or nipple to be treated.

In cases where the above change in the existing equipment cannot be made, the hydrant nipples can be equipped with adapters provided with spanner lugs and having a Standard male thread on the out-board end.

MARKING

Hose.

27. Beginning at a point approximately 4 ft. from the couplings, each 50-ft. length of hose shall be stenciled at two places in black indelible letters 1 in. high with the legend "A.S.T.M. Specifications,"

the name of the manufacturer, the month and year of manufacture and the words "Tested at 400 lb."

28. Both male and female couplings shall be stamped with the name of the manufacturer and the month and year of manufacture in letters and figures not less than $\frac{1}{8}$ in. high. **Couplings.**

INSPECTION AND REJECTION

29. (*a*) The manufacturer shall notify the purchaser sufficiently in advance of the completion of the hose to permit of arrangement for inspection. **Inspection.**

(*b*) The manufacturer shall afford the inspector representing the purchaser, without charge, all reasonable facilities to satisfy him that the hose is being furnished in accordance with these specifications. All tests and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

(*c*) The purchaser may make the tests to govern the acceptance or rejection of the hose in his own laboratory or elsewhere. Such tests, however, shall be made at the expense of the purchaser.

30. (*a*) Each length of hose that fails in the tests specified in Sections 10 (*a*), 11, 12, or 13 or any other requirement of these specifications shall be rejected. **Rejection.**

(*b*) In the case of failure to pass any one of the tests specified in Sections 8, 9, 10 (*b*) or 10 (*c*), the entire lot of hose represented by the sample, or samples, subjected to these tests shall be rejected.

31. Samples of rejected hose shall be preserved for two weeks from the date of test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time. **Rehearing.**

STANDARD SPECIFICATIONS
FOR
WRAPPED AIR HOSE FOR USE WITH PNEUMATIC TOOLS[1]

Serial Designation: D 46 – 24

These specifications are issued under the fixed designation D 46; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1917; ADOPTED, 1918; REISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1924.

Scope.

1. These specifications cover wrapped air hose for use with pneumatic drills, hammers, hoists and other devices working at not more than 125-lb. pressure and for vacuum cleaning outfits.

MANUFACTURE

Construction.

2. The hose shall consist of:

(*a*) An inner rubber tube;

(*b*) Reinforcement of plies of cotton duck;

(*c*) An outer rubber cover;

(*d*) A protective covering of wire armor or a woven cotton jacket when specified.

Rubber Tube.

3. The rubber tube shall be smooth, uniform in quality and thickness, and free from injurious defects.

Cotton Reinforcement.

4. (*a*) The reinforcement shall consist of plies of cotton duck cut on a bias of approximately 45 deg., and applied evenly and firmly over the tube with not less than a $\frac{1}{2}$-in. lap. The number of plies shall be as specified in Table I. The plies of duck shall be well frictioned on both sides with a rubber compound which shall firmly join the plies to the rubber tube and cover and to each other.

(*b*) The cotton shall be as free from unsightly defects, dirt, knots, lumps and irregularities of twist as is consistent with the best manufacturing practice.

(*c*) The reinforcement shall be sufficiently strong to enable the hose to successfully withstand the hydrostatic pressure test and yet at the same time be soft and pliable.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

TABLE I.—NUMBER OF PLIES.

INSIDE DIAMETER OF HOSE, IN.	NUMBER OF PLIES.
$\frac{3}{8}$, $\frac{7}{16}$, $\frac{1}{2}$, $\frac{5}{8}$, $\frac{3}{4}$	4
1, $1\frac{1}{8}$, $1\frac{1}{4}$, $1\frac{1}{2}$	5

5. The rubber cover shall be uniform in quality and thickness and free from injurious defects. **Rubber Cover.**

6. The extra coverings of wire armor or woven cotton jacket when specified shall be as follows: **Extra Coverings.**

(*a*) For hose 1 in. or less in diameter, the wire armor shall be $\frac{5}{32}$ by $\frac{1}{16}$-in. half-elliptical, galvanized mild steel wire wrapped with a maximum space of $\frac{3}{8}$ in. between adjacent turns. For hose over 1 in. in diameter, the wire armor shall be $\frac{3}{16}$ by $\frac{3}{32}$-in. half-round, galvanized mild steel wire wrapped with a maximum space of $\frac{3}{8}$ in. between adjacent turns. Any other size of half-elliptical or half-round wire of equal or greater area may be used provided neither dimension varies more than $\frac{1}{64}$ in. from that specified above.

(*b*) The weight of the woven cotton jacket per linear foot shall be not less than the following:

SIZE OF HOSE, IN.	MINIMUM WEIGHT, OZ. PER LIN. FT.
$\frac{3}{8}$	$\frac{3}{4}$
$\frac{7}{16}$	$\frac{7}{8}$
$\frac{1}{2}$	1
$\frac{5}{8}$	$1\frac{1}{4}$
$\frac{3}{4}$	$1\frac{1}{2}$
1	2
$1\frac{1}{8}$	$2\frac{1}{4}$
$1\frac{1}{4}$	$2\frac{1}{2}$
$1\frac{1}{2}$	3

PHYSICAL PROPERTIES AND TESTS

7. The tests necessary to determine the physical properties which are prescribed in the following sections shall be made in accordance with the Standard Methods of Testing Rubber Products (Serial Designation: D 15) of the American Society for Testing Materials.[1] **Standard Methods.**

8. A sample 36 in. in length shall be cut from each lot of 500 ft. of hose or less and subjected to the tests described in Sections 9, 10 and 11. **Test Sample.**

9. (*a*) In the case of hose 1 in. or less in diameter the test specimens shall be cut longitudinally from the sample of the hose; in the case of hose over 1 in. in diameter the test specimens shall be cut transversely. **Tests of Tube and Cover.**

[1] See p. 847.

(*b*) The tensile strength shall be not less than 800 lb. per sq. in. for the tube and 1000 lb. per sq. in. for the cover.

(*c*) The elongation at the breaking load shall be such that the original 2-in. gage length of the test specimens shall stretch to not less than 7 in. in the case of the tube and 9 in. in the case of the cover.

(*d*) The set, or permanent elongation, following a stretch from 2 in. to 5 in. in the case of the tube and from 2 in. to 7 in. in the case of the cover shall not exceed 25 per cent.

Friction Test. 10. The adhesion between plies shall be such that a weight of 18 lb. will not cause separation at a rate greater than 1 in. per minute.

TABLE II.—TEST PRESSURES.

INSIDE DIAMETER OF HOSE, IN.	TEST PRESSURE, LB. PER SQ. IN.
$\frac{3}{8}$	700
$\frac{7}{16}$	675
$\frac{1}{2}$	650
$\frac{5}{8}$	625
$\frac{3}{4}$	625
1	600
$1\frac{1}{8}$	575
$1\frac{1}{4}$	575
$1\frac{1}{2}$	550

The adhesion between the cover and plies and also between the tube and the plies shall be such that a weight of 15 lb. will not cause separation at a rate greater than 1 in. per minute.

Hydrostatic Pressure Tests. 11. From the 36-in. sample, a piece of hose 24 in. in length shall be taken and subjected to a hydrostatic test at the pressure specified in Table II for hose of its size.

The test shall continue for five minutes and the hose shall withstand the pressure without bursting or developing leaks or other defects. The wire protective covering shall be removed from protected hose prior to this test.

On large orders the number of tests may be reduced to not less than three at the option of the inspector representing the purchaser.

STANDARD SIZES AND DIMENSIONS

Length. 12. Unless otherwise specified, the hose shall be furnished in 50-ft. lengths. The ends of each length shall be uncapped and without fittings.

Size and Thickness. 13. The minimum thicknesses shall be $\frac{3}{32}$ in. for the rubber tube and $\frac{1}{16}$ in. for the rubber cover.

Tolerances, Inside Diameter. 14. The inside diameter shall vary not more than $\pm\frac{1}{64}$ in. from that specified in the case of $\frac{3}{8}$ to $\frac{3}{4}$-in. hose, inclusive, nor more than $\pm\frac{1}{32}$ in. from that specified for larger sizes.

MARKING

15. Each 50-ft. length of hose shall have inlaid in the rubber cover in three places, namely, midway between the ends and within 10 ft. of each end, a brand showing the name of the manufacturer, the month and year of manufacture, the trade name of the hose, and the legend "Wrapped-Air 125 lb. A.S.T.M. Specifications." The letters and figures shall be $\frac{1}{4}$ in. high, shall stand $\frac{1}{32}$ in. in relief, and shall be clear and distinct. **Marking.**

INSPECTION AND REJECTION

16. (*a*) The manufacturer shall notify the purchaser sufficiently in advance of the completion of the hose to permit of arrangement for inspection. **Inspection.**

(*b*) The manufacturer shall afford the inspector representing the purchaser, without charge, all reasonable facilities to satisfy him that the hose is being furnished in accordance with these specifications. All tests and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

(*c*) The purchaser may make the tests to govern the acceptance or rejection of the hose in his own laboratory or elsewhere. But such tests shall be made within 60 days after date of shipment and at the expense of the purchaser.

17. (*a*) Each length of hose that fails to meet any of the requirements of these specifications shall be rejected. **Rejection.**

(*b*) In the case of failure to pass any one of the tests specified in Sections 9, 10, and 11, the entire lot of hose represented by the sample, or samples, subjected to these tests shall be rejected.

18. Samples of rejected hose shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the test, the manufacturer may make claim for a rehearing within that time. **Rehearing.**

STANDARD SPECIFICATIONS
FOR
BRAIDED AIR HOSE FOR USE WITH PNEUMATIC TOOLS[1]

Serial Designation: D 60 – 24

These specifications are issued under the fixed designation D 60; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1919; ADOPTED, 1920; REISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1924.

Scope.

1. These specifications cover braided air hose for use with pneumatic hammers, drills, hoists and other devices working at not more than 125-lb. pressure, and for vacuum cleaning outfits.

MANUFACTURE

Construction.

2. The hose shall consist of:

(*a*) An inner rubber tube;

(*b*) Reinforcement of plies of braided cotton;

(*c*) An outer rubber cover;

(*d*) A protective covering of wire armor or a woven cotton jacket when specified.

Rubber Tube.

3. The rubber tube shall be smooth, uniform in quality and thickness, and free from injurious defects.

Cotton Reinforcement.

4. (*a*) The reinforcement shall consist of plies of braided cotton evenly and firmly woven over the rubber tube—two plies for $\frac{1}{2}$-in. hose and smaller and three plies for $\frac{5}{8}$-in. hose and larger. The plies of braid shall be well impregnated with a rubber compound which shall firmly join the plies to the rubber tube and rubber cover and to each other. There shall be a sufficient coating of rubber compound between the plies so as to form a distinct layer of rubber.

(*b*) The cotton shall be as free from unsightly defects, dirt, knots, lumps and irregularities of twist as is consistent with the best manufacturing practice.

(*c*) The reinforcement shall be sufficiently strong to enable the hose to successfully withstand the hydrostatic pressure test, yet at the same time be soft and pliable.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

5. The rubber cover shall be uniform in quality and thickness, and free from injurious defects. Rubber Cover.

6. When extra coverings of wire armor or braided cotton are specified, they shall be applied as follows: Extra Coverings.

(*a*) For hose 1 in. or less in diameter, the wire armor shall be $\frac{5}{32}$ by $\frac{1}{16}$-in. half-elliptical, galvanized mild steel wire wrapped with a maximum space of $\frac{3}{8}$ in. between adjacent turns. For hose over 1 in. in diameter, the wire armor shall be $\frac{3}{16}$ by $\frac{3}{32}$-in. half-round, galvanized mild steel wire wrapped with a maximum space of $\frac{3}{8}$ in. between adjacent turns. Any other size of half-elliptical or half-round wire of equal or greater area may be used provided neither dimension varies more than $\frac{1}{64}$ in. from that specified above.

(*b*) The weight of the woven cotton jacket per linear foot shall be not less than the following values:

SIZE OF HOSE, IN.	MINIMUM WEIGHT, OZ. PER LIN. FT.
$\frac{3}{8}$	$\frac{3}{4}$
$\frac{7}{16}$	$\frac{7}{8}$
$\frac{1}{2}$	1
$\frac{5}{8}$	$1\frac{1}{4}$
$\frac{3}{4}$	$1\frac{1}{2}$
1	2
$1\frac{1}{8}$	$2\frac{1}{4}$
$1\frac{1}{4}$	$2\frac{1}{2}$
$1\frac{1}{2}$	3

PHYSICAL PROPERTIES AND TESTS

7. The tests necessary to determine the physical properties which are prescribed in the following sections shall be made in accordance with the Standard Methods of Testing Rubber Products (Serial Designation: D 15) of the American Society for Testing Materials.[1] Standard Methods.

8. A sample 36 in. in length shall be cut from each lot of 500 ft. of hose or less and subjected to the tests described in Sections 9, 10 and 11. Test Sample.

9. (*a*) In the case of hose 1 in. or less in diameter, the test specimens shall be cut longitudinally from the sample of the hose; in the case of hose over 1 in. in diameter, the test specimens shall be cut transversely. Tests of Tube and Cover.

(*b*) The tensile strength shall be not less than 800 lb. per sq. in. for the tube of 1000 lb. per sq. in for the cover.

(*c*) The elongation at the breaking load shall be such that the original 2-in. gage length of the test specimens shall stretch to not less than 7 in. in the case of the tube and 9 in. in the case of the cover.

[1] See p. 847.

(*d*) The set, or permanent elongation following a stretch from 2 in. to 5 in. in the case of the tube and from 2 in. to 7 in. in the case of the cover shall not exceed 25 per cent.

Friction Test.

10. The adhesion between plies shall be such that a weight of 18 lb. will not cause separation at a rate greater than 1 in. per minute. The adhesion between the cover and plies and also between the tube and the plies shall be such that a weight of 15 lb. will not cause separation at a rate greater than 1 in. per minute.

Hydrostatic Test.

11. From the 36-in. sample described in Section 8, a piece of hose 24 in. in length shall be taken and subjected to a hydrostatic test at the pressure specified in Table I for hose of its size.

The test shall continue for five minutes and the hose shall withstand the pressure without bursting or developing leaks or other defects. The protective covering shall be removed from protected hose prior to this test.

On large orders the number of tests may be reduced to not less than three at the option of the inspector representing the purchaser.

TABLE I.—TEST PRESSURES.

INSIDE DIAMETER OF HOSE, IN.	TEST PRESSURE, LB. PER SQ. IN.
$\frac{3}{8}$	800
$\frac{7}{16}$	800
$\frac{1}{2}$	800
$\frac{5}{8}$	800
$\frac{3}{4}$	800
1	700
$1\frac{1}{8}$	650
$1\frac{1}{4}$	600
$1\frac{1}{2}$	550

STANDARD DIMENSIONS AND WEIGHTS

Length.

12. Unless otherwise specified, the hose shall be furnished in 50-ft. lengths. The ends of each length shall be uncapped and without fittings.

Thickness.

13. (*a*) The minimum thickness for the tube of $\frac{3}{8}$ and $\frac{7}{16}$-in. hose shall be 0.045 in.; for $\frac{1}{2}$ to $\frac{3}{4}$-in. hose, inclusive, the minimum thickness for the tube shall be 0.062 in., and for 1 to $1\frac{1}{2}$-in. hose, inclusive, the minimum thickness of the tube shall be 0.094 in. The minimum thickness for the cover of $\frac{3}{8}$ and $\frac{7}{16}$-in. hose shall be 0.045 in.; for $\frac{1}{2}$ to $1\frac{1}{2}$-in. hose, inclusive, the minimum thickness of the cover shall be 0.062 in.

(*b*) The gage of the tube and cover shall be measured with the braid embedded in the rubber.

14. The inside diameter shall vary not more than $\pm\frac{1}{32}$ in. from that specified in the case of $\frac{3}{8}$ to $\frac{3}{4}$-in. hose, inclusive, nor more than $\pm\frac{1}{16}$ in. from that specified for larger sizes. Tolerances.

MARKING

15. Each 50-ft. length of hose shall have inlaid in the rubber cover in three places, namely, midway between the ends and within 10 ft. of each end, a brand showing the name of the manufacturer, the month and year of manufacture, the trade name of the hose and the legend "Braided-Air 125 lb. A.S.T.M. Specifications." The letters and figures shall be $\frac{1}{4}$ in. high, shall stand $\frac{1}{32}$ in. in relief, and shall be clear and distinct. Marking.

INSPECTION AND REJECTION

16. (*a*) The manufacturer shall notify the purchaser sufficiently in advance of the completion of the hose to permit of arrangement for inspection. Inspection.

(*b*) The manufacturer shall afford the inspector representing the purchaser, without charge, all reasonable facilities to satisfy him that the hose is being furnished in accordance with these specifications. All tests and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

(*c*) The purchaser may make the tests to govern the acceptance or rejection of the hose in his own laboratory or elsewhere. Such tests shall be made within 60 days after date of shipment and at the expense of the purchaser.

17. (*a*) Each length of hose that fails to meet any of the requirements of these specifications shall be rejected. Rejection.

(*b*) In the case of failure to pass any one of the tests specified in Sections 9, 10, and 11, the entire lot of hose represented by the sample, or samples, subjected to these tests shall be rejected.

18. Samples of rejected hose shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time. Rehearing.

STANDARD SPECIFICATIONS
FOR
WRAPPED COLD WATER HOSE[1]

Serial Designation: D 177 – 24

These specifications are issued under the fixed designation D 177; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED, 1924.

Scope.

1. These specifications cover hose for use in conveying cold water for other than public or private fire department use.

MANUFACTURE

Construction.

2. The hose shall consist of:

(*a*) An inner rubber tube;

(*b*) Reinforcement of plies of cotton duck;

(*c*) An outer rubber cover.

Rubber Tube.

3. The rubber tube shall be smooth, uniform in quality and thickness and free from injurious defects.

Cotton Reinforcement.

4. (*a*) The reinforcement shall consist of plies of cotton duck cut on a bias of approximately 45 deg., and applied evenly and firmly over the tube with not less than a $\frac{1}{2}$-in. lap, not sewed. The number of plies shall be as follows:

INSIDE DIAMETER OF HOSE, IN.	NUMBER OF PLIES
$\frac{1}{2}$, $\frac{3}{4}$	3
1, $1\frac{1}{4}$, $1\frac{1}{2}$, 2, $2\frac{1}{4}$	4
$2\frac{1}{2}$ to 4	5

The plies of duck shall be well frictioned on both sides with a rubber compound which shall firmly join the plies to the rubber tube and cover and to each other.

(*b*) The duck shall be evenly woven from high grade cotton and shall be free from mechanical defects.

(*c*) The reinforcement shall be sufficiently strong to enable the hose to successfully withstand the hydrostatic pressure test and yet at the same time be soft and pliable.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

5. The rubber cover shall be uniform in quality and thickness, and be free from injurious defects. Rubber Cover.

PHYSICAL PROPERTIES AND TESTS

6. The tests necessary to determine the physical properties prescribed in the following sections shall be made in accordance with the Standard Methods of Testing Rubber Products (Serial Designation: D 15) of the American Society for Testing Materials.[1] Standard Methods.

7. A sample 36 in. in length shall be cut from each lot of 500 ft. or fraction thereof of each size of hose. Test Sample.

8. (*a*) In the case of hose 1 in. or less in diameter, the test specimens shall be cut longitudinally from the sample of the hose; in the case of hose over 1 in. in diameter, the test specimens shall be cut transversely. Tests of Tube and Cover.

(*b*) The tensile strength shall be not less than 600 lb. per sq. in. for the tube and 450 lb. per sq. in. for the cover.

(*c*) The elongation at the breaking load shall be such that the original 2-in. gage length of the test specimens shall stretch to not less than 8 in. in the case of the tube and 6 in. in the case of the cover.

(*d*) The set or permanent elongation following a stretch from 2 in. to 6 in. in the case of the tube and from 2 in. to 4 in. in the case of the cover shall not exceed 25 per cent.

9. The adhesion between plies shall be such that a weight of 15 lb. will not cause separation at a rate greater than 1 in. per minute. The adhesion between the cover and the plies, and also between the tube and the plies shall be such that a weight of 12 lb. will not cause separation at a rate greater than 1 in. per minute. Friction Test.

10. The remainder of the 36-in. sample after all other test specimens have been cut therefrom shall withstand a hydrostatic pressure test as follows: Hydrostatic Pressure Test.

Inside Diameter of Hose, in.	Test Pressure, lb. per sq. in.
¼, ⅜	450
1, 1¼, 1½	350
2, 2¼, 2½	300
3, 3½ and 4	250

STANDARD SIZES AND DIMENSIONS

11. Unless otherwise specified, the hose shall be furnished in 50-ft. lengths. The ends of each length shall be uncapped and without fittings. Length.

12. The minimum thickness for the tube of all sizes of hose shall be 0.062 in. The minimum thickness for the cover for ½ and ¾-in. Thickness of Tube and Cover.

[1] See p. 847.

hose shall be 0.031 in. and for all other sizes of hose, the minimum thickness for the cover shall be 0.047 in.

Inside Diameter.

13. The inside diameter shall not vary more than $\pm\frac{1}{64}$ in. from that specified for $\frac{1}{2}$ and $\frac{3}{4}$-in. hose, nor more than $\pm\frac{1}{32}$ in. from that specified for all other sizes.

MARKING

Marking.

14. Each length of hose 25 ft. or over in length shall have a brand inlaid in the rubber cover in three places: namely, approximately in the center and approximately 5 ft. from each end. Each length of less than 25 ft. shall have a brand inlaid approximately in the center. The brand shall show the name of the manufacturer, the kind and size of hose, the month and year of manufacture, the trade name of the hose, and the legend "Wrapped Cold Water, ... lb., A.S.T.M. Specifications." The letters and figures shall be $\frac{1}{4}$ in. high, $\frac{1}{32}$ in. in relief and shall be clear and distinct.

INSPECTION AND REJECTION

Inspection.

15. (*a*) The manufacturer shall notify the purchaser sufficiently in advance of the completion of the hose to permit of arrangement for inspection.

(*b*) The manufacturer shall afford the inspector representing the purchaser, without charge, all reasonable facilities to satisfy him that the hose is being furnished in accordance with these specifications. All tests and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

(*c*) The purchaser may make the tests to govern the acceptance or rejection of the hose in his own laboratory or elsewhere. But such tests shall be made within 60 days after date of shipment and at the expense of the purchaser.

Rejection.

16. (*a*) Any length which fails to meet any of the requirements of these specifications shall be rejected.

(*b*) In the case of failure to pass any one of the tests specified in Sections 8, 9 and 10, the entire lot of hose represented by the sample, or samples, subjected to these tests shall be rejected.

Rehearing.

17. Samples of rejected hose shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the test, the manufacturer may make claim for a rehearing within that time.

STANDARD SPECIFICATIONS
FOR
RUBBER BELTING FOR POWER TRANSMISSION[1]

Serial Designation: D 53 - 24

These specifications are issued under the fixed designation D 53; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1918; ADOPTED IN AMENDED FORM, 1920; REISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1924.

Scope.

1. These specifications cover two classes of rubber belting for power transmission:

(*a*) *Friction Covered.*—Possessing no distinct rubber cover other than that imparted to the cotton duck by the regular rubber frictioning process.

(*b*) *Rubber-Covered.*—Possessing a distinct rubber cover over the outside plies of rubber-frictioned cotton duck and extending around the edges.

MANUFACTURE

Construction.

2. The belting shall consist of:

(*a*) Cotton duck reinforcement;

(*b*) Rubber friction compound;

(*c*) An outside rubber cover, when specified.

Reinforcement.

3. The reinforcement shall consist of plies of cotton duck evenly and firmly woven, as free from such defects as foreign matter, knots, lumps and irregularities of twist as is consistent with the best manufacturing practice. Each ply shall be well frictioned on both sides with a rubber compound suitable for the required service.

Rubber Beading.

4. The seam in the outside ply of the belting shall be completely filled with a rubber cord, or "beading," well fastened down with a rubber cover-strip.

Rubber Cover.

5. When rubber-covered belting is specified, the rubber cover shall be uniform in thickness and free from cracks, blisters or other defects which would impair its moisture-resisting qualities.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

PHYSICAL PROPERTIES AND TESTS

Temperature of Testing Room and Samples.

6. Tests shall be made with the temperature of the air not lower than 65° F. or higher than 90° F., and the samples shall be kept at temperatures within these limits for at least 30 minutes previous to the time of test.

Sampling.

7. One sample, the full width of the belt, shall be taken from each lot of 500 ft. or less. This sample shall be 26 in. in length for all widths 6 in. and over, and 32 in. for widths under 6 in.

Number of Tests.

8. One friction test, one stretch test, one tension test, one beading test and three filler strength tests shall be made upon the sample described in Section 7.

Test Specimens.

9. Friction specimens may be taken either longitudinally or transversely of the sample of belting. Stretch and tension test specimens shall be the full width and thickness of the belting up to and including 5 in. For widths greater than 5 in. the test specimen shall be cut to a width of 5 in., parallel to an edge and beginning $\frac{1}{2}$ in. from one of the edges of the belting.

Friction Test.

10. (*a*) Tests to determine the adhesion between plies shall be made in accordance with the Standard Methods of Testing Rubber Products (Serial Designation: D 15) of the American Society for Testing Materials.[1]

(*b*) The adhesion between the plies shall be such that a weight of 20 lb. will not cause separation at a rate greater than 1 in. per minute.

Stretch Test of Belting.

11. A test specimen 20 in. in length and prepared as described in Section 9 shall have transverse reference lines marked upon it 10 in. apart and at equal distances from the ends; it shall then be tested in a tension testing machine and under a pull of 100 lb. per in. per ply, shall stretch not more than 9 per cent nor less than 5 per cent. The rate of separation of the jaws shall be approximately $1\frac{1}{2}$ in. to 4 in. per minute.

Tension Test.

12. The stretch test described in Section 11 shall be continued to the breaking point in order to obtain the ultimate tensile strength which shall be not less than 260 lb. per in. per ply.

Test of Beading.

13. The beading in all belting 4 in. wide or over shall be tested as follows: A 4-in. transverse section of belting shall be taken and beginning on the seamless side, the plies shall be removed until three only remain intact. A line shall be drawn down the middle of the beading and then on each side of this line, and $\frac{5}{8}$ in. distant from it, two parallel lines shall be scribed. The section shall be inserted in a vise so that the jaws grip the sample coincident with the two outer

[1] See p. 847.

lines. The vise shall be tightened until the inner surfaces of the doubled sample just touch at the top of the vise, and the sample shall be so held for 10 minutes. Under this test the beading shall not crack or loosen in the seam.

Test of Filler Strength of Duck.

14. The filler or weft strength of the cotton duck in the belting shall be determined as follows: A transverse strip $1\frac{1}{2}$ in. wide shall be cut from the belting and separated into its various plies. Three of these plies shall be ravelled down to a width of 1 in. and tested in a fabric testing machine, the minimum width of jaws of which is $1\frac{1}{2}$ in., the distance between jaws 3 in. and the speed of separation of jaws approximately 20 in. per minute. The tensile strength shall be at least 175 lb.

DIMENSIONS

Width of Belting.

15. The width of the belting shall conform to that specified within the following tolerances:

WIDTH.	PERMISSIBLE PLUS OR MINUS VARIATIONS, IN.
Up to 12 in., inclusive	$\frac{3}{16}$
Over 12 in. to 24 in., inclusive	$\frac{1}{4}$
Over 24 in.	$\frac{5}{16}$

Thickness of Belting.

16. The thickness of the belting in inches shall be not less than 0.050 multiplied by the number of plies, plus the thickness of the rubber covers, if any.

Thickness of Cover.

17. The cover (Section 1 (*b*)) shall be not less than $\frac{3}{128}$ (=0.023) in. in thickness. This shall also apply to the edges.

MARKING

Marking.

18. Belting 4 in. wide or over shall have the name of the manufacturer, the month and year of manufacture, the trade name of the belt, and the legend "Power A.S.T.M. Specifications" impressed in the strip side at points not more than 15 ft. apart. For widths under 4 in. this information shall be stencilled on the belting.

Endless Belts.

19. Endless belts shall have marks showing in which direction the belt shall be run with regard to the splices.

INSPECTION AND REJECTION

Inspection.

20. (*a*) The manufacturer shall notify the purchaser sufficiently in advance of the completion of the belting to permit of arrangement for inspection.

(*b*) The manufacturer shall afford the inspector representing the purchaser, without charge, all reasonable facilities to satisfy him that the belting is being furnished in accordance with these specifica-

tions. All tests and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

(*c*) The purchaser may make the tests to govern the acceptance or rejection of the belting in his own laboratory or elsewhere. Such tests shall be made within 60 days after date of shipment at the expense of the purchaser.

Rejection. 21. Any length of belting which fails to meet the requirements of these specifications shall be rejected.

Rehearing. 22. Samples of rejected belting shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

STANDARD SPECIFICATIONS
FOR
RUBBER GLOVES FOR ELECTRICAL WORKERS ON APPARATUS OR CIRCUITS NOT EXCEEDING 3000 VOLTS TO GROUND[1]

Serial Designation: D 120 – 23

These specifications are issued under the fixed designation D 120; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1923.

SPECIFICATIONS.

Scope.

1. These specifications cover electrical workers' rubber gloves of two classes:

Class A.—Gloves intended for use without external protection.

Class B.—Gloves intended for use with external protection of leather or other material.

MANUFACTURE

Process.

2. The gloves shall be made by the "dipping" process, of high-grade rubber or rubber compound properly vulcanized, and shall be reversible. Unless otherwise specified cuff edges shall be finished with a roll.

PHYSICAL PROPERTIES AND TESTS

Methods.

3. All tests shall be made in accordance with the methods described in Sections 15 to 26, inclusive.

(*A*) *Electrical.*

Voltage and Leakage Tests.

4. (*a*) Each glove shall withstand an alternating potential of 10,000 volts (r.m.s. value) for three minutes without showing a leakage current of more than 10 milliamperes (total current) at any time during the test.

(*b*) Each glove shall be capable of withstanding the application of the following voltage (r.m.s. value) for one minute without showing a leakage current in excess of that given below:

[1]Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

	Volts.	Milliamperes.
Class A	16 000	18
Class B	14 000	14

(*B*) *Mechanical.*

Tensile Strength, Elongation and Set.

5. A standard test specimen cut from any glove shall have the following physical properties:

(*a*) The tensile strength shall be not less than 1200 lb. per sq. in.

(*b*) The total elongation at rupture shall be not less than 500 per cent in 2 in. (2 in. stretched to 12 in.).

(*c*) The set following a stretch from 2 to 12 in., shall not exceed 0.7 in.

STANDARD SIZES AND DIMENSIONS

Size and Length.

6. (*a*) The size of each glove shall be as specified subject to a permissible variation of $\pm\frac{1}{4}$ in. and shall be expressed in inches as measured around the palm at the base of knuckles.

(*b*) Unless otherwise specified the length of each glove from the tip of the second finger to the edge of cuff, shall be 14 in. subject to a permissible variation of $\pm\frac{1}{2}$ in.

Thickness.

7. The respective thicknesses of the two classes of gloves in inches shall be within the following limits:

	Part of Glove Other than Crotch.		Crotch.	
	Maximum.	Minimum.	Maximum.	Minimum.
Class A	0.065	0.050	0.065	0.038
Class B	0.050	0.030	0.050	0.022

WORKMANSHIP AND FINISH.

Surface Defects.

8. Gloves shall be free from patches, blisters, pinholes, cracks, protuberances, embedded foreign matter or other physical defects which can be detected by thorough surface inspection.

MARKING AND PACKING

Marking.

9. Unless otherwise specified each glove shall be marked with the manufacturer's name and the size of glove. Such marking shall be confined to the back of the glove and shall be so done as not to injure the glove. Indelible and non-conducting ink shall be used.

Packing.

10. Each pair of gloves shall be packed in an individual stiff paper box of sufficient strength to properly protect the gloves. The end of the box shall be marked with the glove size and in the case of Class B gloves with the words "For use with Protectors."

GUARANTEE

Guarantee.

11. The manufacturer shall replace, without cost to the purchaser, gloves which at any time within a period of eight months from the date

of initial test, fail to pass the tests herein prescribed; provided the gloves have been properly stored in their original boxes.

NOTE.—"Proper storage" is to be interpreted to mean that gloves are not to be stored directly above or in proximity to steam radiators or other sources of artificial heat. It is desirable that the ambient temperature be not in excess of 90° F. (32.2° C.).

INSPECTION AND REJECTION

Place and Time of Tests.

12. Inspection and test unless otherwise specified shall be made within two weeks of receipt by the purchaser at his own expense at such place as he may designate.

Rejections.

13. (*a*) Each glove that fails to meet the requirements of Sections 4 (*a*) or 8 shall be rejected. In the case of gloves failing to meet the requirements of Sections 6, 9 and 10 rejection is optional with the purchaser.

(*b*) If any of the measurements of thickness made as prescribed in Section 16 or any additional measurements which the purchaser may elect to make fall below the minimum thickness specified in Section 7, the glove shall be rejected. If any of the measurements exceed the maximum thickness specified, rejection is optional with the purchaser.

(*c*) If rejections under Paragraphs (*a*) and (*b*) exceed 25 per cent of the shipment the whole shipment may be rejected without further tests.

(*d*) If 50 per cent or more of the gloves subjected to the test prescribed in Section 4 (*b*) fail to pass the test, the entire shipment shall be rejected.

(*e*) If the average of the results for either the tensile strength, elongation or set tests made as prescribed in Section 26 fail to meet the requirements of Section 5, the entire shipment shall be rejected.

Marking Rejected Gloves.

14. All rejected material shall be returned to the manufacturer without being defaced by rubber stamp or other permanent marking, excepting gloves which have been punctured on electrical tests. These shall be stamped, punched or cut to indicate that they are unfit for electrical use.

METHODS OF TEST

Order of Procedure.

15. The order of procedure in testing rubber gloves shall preferably be as follows:[1]

(*a*) Inspection of inside and outside surfaces (see Section 8).

(*b*) Measurement of length (see Section 6 (*b*)).

(*c*) Measurement of size (see Section 6 (*a*)).

[1] This procedure has been found by experience to minimize the expense of the inspection and testing, particularly where an appreciable number of gloves fail to meet the requirements as to workmanship or dimensions.

(*d*) Measurement of thickness (see Section 7).
(*e*) Electrical test (see Section 4 (*a*)).
(*f*) Dielectric strength (see Section 4 (*b*)).
(*g*) Mechanical test (see Section 5).

DIMENSIONS

Thickness. 16. (*a*) The thickness of each glove shall be measured at not less than four points on the face and four points on the back.

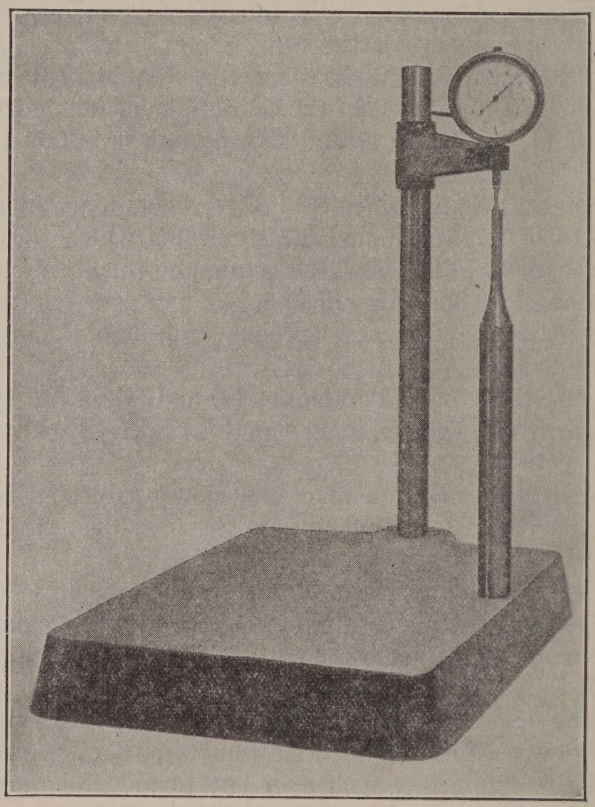

FIG. 1.—Dial Micrometer.

(*b*) Measurements shall be made at one or more points in the crotch of thumb and index finger and in the crotches between the fingers.

(*c*) These measurements shall be made with any micrometer graduated to mils or tenths of mils having a ratchet friction or spring attachment. The tension on the friction attachment should be so adjusted that when the spindle has come to a stop, the specimen can

be moved between the spindle and anvil without causing any appreciable stretching of the rubber.

A dial-type micrometer graduated to mils and mounted in a manner similar to that shown in Fig. 1 is particularly convenient for making all of these measurements, especially the crotches. The anvil is about $\frac{1}{4}$ in. (6.4 mm.) in diameter and the foot of the spindle about $\frac{1}{8}$ in. (3.2 mm.) in diameter.

ELECTRICAL TESTS

Leakage.

17. Each glove shall be tested for leakage in fresh water at normal room temperature (60 to 90° F.) by immersing in the water to within about $1\frac{1}{2}$ to 2 in. of the edge of the cuff and filling with water up to the same point. The water inside the glove and that outside the glove form the test electrodes. These are conveniently connected to the testing transformer by means of a chain suspended in the glove and by direct connection to the containing vessel.

Source of Test Voltage.

18. The testing voltage shall be obtained from a testing equipment, no part of which has a testing capacity of less than $\frac{1}{2}$ kva. per glove being tested. In no case shall the rating of any part of the testing apparatus be less than 2 kva. The frequency of the testing voltage shall be not more than 65 cycles.

Application of Test Voltage.

19. The potential shall be applied at a low value and gradually and steadily raised at a rate of approximately 800 to 1000 volts per second until the prescribed testing voltage is reached.

Test Period.

20. The test period shall be counted from the instant when the prescribed testing voltage is reached.

Voltage Regulation.

21. The method of regulating the testing voltage shall be one which does not distort the wave form of the testing voltage from a sine wave. Acceptable methods include:

(*a*) Field regulation of the alternator supplying the transformer;

(*b*) Induction-type regulator;

(*c*) Variable-ratio-transformer type of regulator;

(*d*) Potentiometer type of rheostatic control where the current in the portion of the potentiometer resistance in parallel with the primary of the transformer is at least five times the exciting current of the transformer.

Voltage Measurement.

22. The testing voltage shall be measured by one of the following methods:

(*a*) A properly calibrated electrostatic voltmeter connected directly across the gloves under test.

(*b*) Any properly calibrated commercial type of alternating current voltmeter connected to the low-tension side of the transformer

in conjunction with the ratio of transformation of the transformer provided that the ratio if definitely known for all test conditions.

(*c*) A calibrated potential transformer with a voltmeter.

Leakage Current Measurement.

23. The leakage current shall be measured by one of the following methods:

(*a*) Directly, by inserting a milliammeter in series with each individual glove in turn.

NOTE.—It is customary to make high voltage tests of this character with one end of the circuit grounded, the containing vessel usually being the grounded end of the circuit. The milliammeter should therefore be connected in at the grounded end of the circuit and shunted by a short circuiting automatic self-closing switch which will therefore always keep the circuit closed except at the instant of reading and thus maintaining an uninterrupted ground.

(*b*) By an ammeter in the low-tension circuit of the testing transformer. In this case the leakage current shall be determined by noting the decrease in the low-tension current when *one glove at a time* is disconnected from the high tension testing circuit, the voltage being left on the test circuit and the remaining gloves in the meantime. The difference in the two currents divided by the ratio will give the leakage current for the glove disconnected from the circuit.

NOTE.—This method is inaccurate where only one or two gloves are being tested at a time because of the neutralizing effect of the leading current taken by the gloves on the magnetizing current, which is a lagging one. This effect is usually negligible with three or more gloves. The minimum number of gloves which may be safely measured in this manner is easily determined by noting the leakage current of a particular glove when tested with 1, 2, 3, etc., additional gloves, respectively.

Dielectric Strength Test.

24. (*a*) One per cent of the gloves (in no case less than two gloves) which have passed all the tests previously prescribed herein shall be selected at random and tested for compliance with Section 4 (*b*) and also for ultimate dielectric strength. The tests shall be made in accordance with the general method described in Sections 17 to 23 inclusive. If the glove passes the test prescribed in 4 (*b*), the potential shall be raised steadily and gradually at a rate of approximately 1000 volts per second until failure occurs, the voltage at failure being recorded.

(*b*) If any glove fails to meet the requirements of Section 4 (*b*), an additional glove shall be selected at random and tested.

MECHANICAL TESTS

Test Specimens.

25. Four or more standard test specimens shall be cut from each glove tested under Section 24. The shape of these specimens shall be in substantial accordance with that prescribed in the Standard

Methods of Testing Rubber Products (Serial Designation: D 15) of the American Society for Testing Materials.[1] The essential dimensions of this sample are:

Total length: 6 in.

Width: 1 or $1\frac{1}{4}$ in. with a reduced section at the center, $\frac{1}{2}$ in. wide and about 2 in. long with radii of curvature between the ends and reduced section of $\frac{7}{8}$ in.

Tensile Strength, Elongation and Set.

26. The tests for tensile strength, elongation and set shall be made in accordance with the Standard Methods of Testing Rubber Products (Serial Designation: D 15) of the American Society for Testing Materials.[1] The essential features of these methods are:

(*a*) Temperature of testing room and of specimens, between 65 and 90° F.

(*b*) Rate of separation of jaws of testing machine, 20 in. per minute.

(*c*) Tensile strength and elongation shall be made on one sample and set test on a separate sample.

(*d*) Set test shall be made by stretching the 2-in. gage length to 12 in., holding for ten minutes and measuring the distance between the bench marks 10 minutes after release.

[1] See p. 847.

STANDARD SPECIFICATIONS FOR RUBBER MATTING FOR USE AROUND ELECTRICAL APPARATUS OR CIRCUITS NOT EXCEEDING 3000 VOLTS TO GROUND[1]

Serial Designation: D 178 - 24

These specifications are issued under the fixed designation D 178; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED, 1924.

SPECIFICATIONS

Scope.

1. These specifications cover rubber matting for use as a floor covering near electrical apparatus.

MANUFACTURE

Construction.

2. The matting shall consist of a rubber compound corrugated on one surface and backed with cotton fabric, or having one or more cotton fabric inserts, the back of the matting being finished with cloth imprint.

PHYSICAL PROPERTIES AND TESTS

(*A*) *Mechanical*

Methods.

3. All the tests necessary to determine the mechanical properties specified in the following sections shall be made on the rubber exclusive of fabric, and shall be made in accordance with Sections 48 to 56, inclusive, of the Standard Methods of Testing Rubber Products (Serial Designation: D 15) of the American Society for Testing Materials.[2]

Sampling.

4. Not less than one sample shall be taken from each piece or roll of matting and subjected to the tests described in Section 5.

Tensile Strength, Elongation and Set.

5. (*a*) The tensile strength shall be not less than 700 lb. per sq. in.

(*b*) The elongation at the breaking load shall be such that the original 2-in. gage length of the test specimens shall stretch to not less than 7 in.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

[2] See p. 847.

(*c*) The set, following a stretch from 2 in. to 5 in., shall not exceed 25 per cent.

(*B*) *Electrical*

Methods. 6. All the tests necessary to determine the electrical properties specified in Sections 7 and 8 shall be made in accordance with the methods described in Sections 13 to 17, inclusive.

Voltage Test. 7. (*a*) The entire area of the matting as nearly as practicable shall be tested between electrodes consisting of rectangular metal sheets, having smoothly rounded edges and corners and of any convenient length, the width being such that arcing around the edges of the matting will not occur, the tests being made progressively until the entire length of the matting has been covered.

(*b*) A potential of 15,000 volts shall be applied for one minute at each position of the electrodes. The matting shall not puncture, become appreciably warm at any spot or show any other sign of weakness.

Dielectric Strength Test. 8. A specimen cut from the sample and tested to failure in air between 2-in. disk electrodes with edges rounded to a radius of $\frac{1}{4}$ in., the voltage being applied at the rate specified in Section 14, shall not fail at less than 40,000 volts.

DIMENSIONS

Thickness. 9. The thickness, unless otherwise specified, shall not be less than $\frac{1}{4}$ in. when measured over the corrugations and not less than $\frac{1}{8}$ in. at the root of the corrugations.

WORKMANSHIP AND FINISH

Surface Defects. 10. Matting shall be free from patches, blisters, pin-holes, cracks, protuberances, embedded foreign matter or other physical defects which can be detected by thorough surface inspection.

INSPECTION AND REJECTION

Inspection. 11. The matting shall be inspected and tested within four weeks of the date of delivery.

Rejection. 12. (*a*) Any piece or roll of matting which fails to pass any of the requirements of these specifications shall be rejected.

(*b*) In case of failure to pass any one of the tests specified in Sections 5 and 8, the piece or roll of matting represented by the sample subjected to these tests shall be rejected.

METHODS OF TESTING

Source of Test Voltage.

13. The testing voltage shall be obtained from a testing equipment, no part of which has a capacity of less than $\frac{1}{2}$ kva. per sq. ft. of electrode surface. In no case shall the rating of any part of the testing apparatus be less than 5 kva. The frequency of the testing voltage shall be not more than 65 cycles.

Application of Test Voltage.

14. The potential shall be applied at a low value and gradually and steadily raised at a rate of approximately 800 to 1000 volts per second until the prescribed testing voltage is reached.

Test Period.

15. The test period shall be counted from the instant when the prescribed testing voltage is reached.

Voltage Regulation.

16. The method of regulating the testing voltage shall be one which does not distort the wave form of the testing voltage from a sine wave. Acceptable methods include:

(*a*) Field regulation of the alternator supplying the transformer;

(*b*) Induction-type regulator;

(*c*) Variable-ratio-transformer type of regulator;

(*d*) Potentiometer type of rheostatic control where the current in the portion of the potentiometer resistance in parallel with the primary of the transformer is at least five times the exciting current of the transformer.

Voltage Measurement.

17. The testing voltage shall be measured by one of the following methods:

(*a*) A properly calibrated electrostatic voltmeter connected directly across the matting under test.

(*b*) Any properly calibrated commercial type of alternating current voltmeter connected to the low tension side of the transformer in conjunction with the ratio of transformation of the transformer provided that the ratio is definitely known for all test conditions.

(*c*) A calibrated potential transformer with a voltmeter.

STANDARD SPECIFICATIONS
FOR
RUBBER PUMP VALVES[1]

Serial Designation: D 151 – 23

These specifications are issued under the fixed designation D 151; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1922; ADOPTED, 1923.

Scope.

1. These specifications cover rubber valves for pumps equipped with grid-type seats and which are used for pumping fluids having very slight, if any, effect on rubber.

Classes.

2. Rubber pump valves shall be classified according to the service for which they are intended as follows:

(*a*) *Class 100.*—Vulcanized hard for hot water at a temperature of 150 to 220° F. and a pressure less than 300 lb. per sq. in.

(*b*) *Class 200.*—Vulcanized medium hard for water at a temperature of less than 170° F. and a pressure less than 350 lb. per sq. in.

(*c*) *Class 300.*—Vulcanized medium soft for water at a temperature less than 170° F. and a pressure less than 200 lb. per sq. in.

(*d*) *Class 400.*—Vulcanized soft for water at a temperature less than 170° F. and a pressure less than 50 lb. per sq. in.

(*e*) *Class 500.*—Vulcanized extra soft for water at a temperature less than 170° F. and a pressure less than 20 lb. per sq. in.

MANUFACTURE

Manufacture.

3. The valves shall consist of properly vulcanized rubber compound and all surfaces shall be smooth and free from pitting, blisters, air checks and other imperfections. The thickness shall be uniform throughout and the circumference shall be concentric with the hole at the center.

CHEMICAL PROPERTIES AND TESTS

Chemical Requirements.

4. (*a*) The compound shall conform to the requirements specified in Table I expressed as percentages, by weight, of the total.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

TABLE I.

CLASS	RUBBER, MINIMUM, PER CENT	SULFUR, MAXIMUM, PER CENT[a] TOTAL	SULFUR, MAXIMUM, PER CENT[a] FREE	ORGANIC ACETONE EXTRACT, MAXIMUM, PER CENT
100	30	7.5	1.0	2.0
200	32	5.0	1.0	2.0
300	35	4.0	1.0	2.0
400	38	3.0	0.75	2.5
500	40	3.0	0.75	2.5

[a] Exclusive of sulfur in barytes.

(*b*) The remainder of the compound shall consist of suitably pulverized inorganic mineral fillers, which may contain barytes, but which shall be practically free from any substance likely to have a deleterious effect on the compound.

(*c*) The compound shall contain no rubber substitute of any kind, rubber which has been previously used or reclaimed rubber.

Chemical Analyses.

5. Chemical analyses of the compound shall be made in accordance with the appropriate section of those numbered from 1 to 14, inclusive, of the Standard Methods of Testing Rubber Products (Serial Designation: D 15) of the American Society for Testing Materials.[1]

PHYSICAL PROPERTIES AND TESTS

Specific Gravity.

6. The specific gravity of the valves shall not exceed the values given in Table II.

Plastometer Readings.

7. (*a*) Plastometer readings shall be taken by noting the depth of identation in millimeters produced by a spherical surface 3.2 mm. (0.125 in.) in diameter under a dead weight pressure of 1 kg. (2.2 lb.) applied for one minute. The test shall be made at a temperature not less than 60° F. nor more than 80° F.

(*b*) The readings obtained on any valve shall not be outside the limits specified in Table II:

TABLE II.

CLASS	PLASTOMETER READING, LIMITS	SPECIFIC GRAVITY, MAXIMUM
100	0.03 – 0.10	1.75
200	0.25 – 0.40	1.70
300	0.55 – 0.70	1.65
400	0.85 – 1.00	1.60
500	1.40 – 1.55	1.60

Heat Tests.

8. No valve, when cut in half and subjected to the following dry heat and steam tests, shall show any disintegration, blistering or other defects either on the surface or in the interior when cut open with a knife:

[1] See p. 847.

(*a*) *Dry heat test.*—One-half of valve at 270° F. ±5° F. dry heat for one hour.

(*b*) *Steam test.*—One-half of valve in steam at 400° F. ±5° F. for three hours, the steam pressure to be gradually reduced to atmospheric pressure at the end of the test.

STANDARD SIZES

Dimensions.

9. The dimensions of the valves shall be as specified by the purchaser.

Permissible Variations.

10. The dimensions (diameter and thickness) shall not vary more than $\frac{1}{32}$ in. from those specified.

MARKING

Marking.

11. Each valve shall be branded on the edge with the letters "A.S.T.M.," the class number, the name of the manufacturer, and the month and year of manufacture.

INSPECTION

Number of Samples.

12. The number of valves selected for inspection and for chemical and physical tests shall be determined by the purchaser.

Inspection and Tests.

13. (*a*) The purchaser may make the inspections and tests to govern the acceptance or rejection of the valves in his own laboratory or elsewhere, but such inspections and tests shall be made at the expense of the purchaser.

(*b*) If the inspections and tests are made at the place of manufacture, the manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the valves are being furnished in accordance with these specifications. Such inspections and tests shall be so conducted as not to interfere unnecessarily with the operation of the works.

STANDARD SPECIFICATIONS
FOR
ADHESIVE TAPE FOR GENERAL USE FOR ELECTRICAL PURPOSES[1]

Serial Designation: D 69 – 24

These specifications are issued under the fixed designation D 69; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1920; ADOPTED IN AMENDED FORM, 1924.

Scope.

1. These specifications cover commercial adhesive tape (also called "friction" tape) consisting of strips of cotton sheeting impregnated with an adhesive insulating compound. The tape is for general use for covering electrical insulation applied to wires and cables.

MANUFACTURE

Cotton Sheeting.

2. The cotton sheeting layer shall be made from a sheeting evenly and firmly woven from good cotton and as free from unsightly defects, dirt, knots, lumps and irregularities of twist as is consistent with the best manufacturing practice. The threads shall run in as straight lines as possible without waving so as to reduce to a minimum the raveling of the cloth when cut into tape.

Frictioning Compound.

3. The frictioning compound shall be an adhesive and insulating compound practically free from free sulfur or other substances which would have a deteriorating effect on copper or other metals or on the fabric.

Impregnation of Fabric.

4. The fabric shall be thoroughly impregnated and evenly covered on both sides with the frictioning compound.

Adhesiveness of Compound.

5. The compound shall adhere firmly to the fabric and shall not pull away from the fabric so as to leave bare spots when adjacent layers of the tape are separated.

CHEMICAL PROPERTIES

Discoloration of Copper.

6. When the tape is wrapped on a clean, bright, smooth copper rod and baked at 100° C. for 16 hours, the copper shall not show the blue-black color characteristic of copper sulfide.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

PHYSICAL PROPERTIES AND TESTS

7. When the tape is held before a strong light, the number of pin holes shall be determined in a length of not less than 3 yd. The number of pin holes shall not exceed the limits given in the following table: **Pin Holes**

WIDTH, IN.	MAXIMUM NUMBER IN 3 YD.	MAXIMUM NUMBER IN ANY SINGLE YD.
½	6	3
¾	6	3
1	9	4
1½	12	5
2	15	6

8. The tensile strength of the tape shall be not less than 40 lb. per inch of width. The initial distance between the jaws of the testing machine shall be 12 in. and the rate of separation of the jaws shall be 20 in. per minute. **Tensile Strength.**

9. (*a*) The adhesion between adjacent layers of the tape shall be such that when a strip of tape 2 ft. long is taken from a roll and wound upon a mandrel 1 in. in diameter under a tension of 10 lb. per inch of width at a rate of 30 in. per minute, and allowed to stand for three minutes with the weight attached, a weight of 4 lb. per inch of width shall not cause the plies to separate at a rate greater than 30 in. per minute. **Friction.**

The test shall be made at a room temperature not less than 21.1° C. (70° F.) nor more than 23.9° C. (75° F.), the sample having been kept within these limits for at least 30 minutes immediately preceding the time of testing.

The mandrel shall be so free in its bearings that a weight of 1 oz. will cause it to revolve freely when suspended from a cotton string wound in a single layer on the center of the mandrel.

(*b*) After a strip has been exposed to dry air at a temperature of not less than 99° C. (210.2° F.) nor more than 101° C. (213.8° F.) for 16 hours and then cooled to room temperature, a test specimen shall withstand the test prescribed in Paragraph (*a*) except that the weight applied to unwind the tape shall be 1⅓ lb. per inch of width instead of 4 lb.

10. The test for dielectric strength shall be made as follows: The tape shall be spirally wound with one-third lap on a smooth metal rod, 1 in. in diameter, for a distance of 6 in. Two inches in the center shall be covered with metal foil and securely bound with tape. Alternating potential of 1000 volts (r.m.s. value) and having a frequency not exceeding 65 cycles per second shall be applied for five **Dielectric Strength.**

minutes to the metal rod and the metal foil without puncturing the tape.

Test Samples.

11. One roll for each 250 rolls shall be taken at random for test. At least 2 ft. of the outer layers shall be removed and discarded before taking specimens for test.

STANDARD WEIGHT, DIMENSIONS AND VARIATIONS

Weights, Dimensions and Yardage.

12. The tape shall conform to the following requirements:

WIDTH, IN.	THICKNESS, IN.	NOMINAL WEIGHT PER ROLL, LB.	MINIMUM NET WEIGHT PER 100 ROLLS, LB.[a]	MINIMUM LENGTH PER POUND, YD.[a]
$\frac{1}{2}$	0.015	$\frac{1}{4}$	25	82
$\frac{3}{4}$	0.015	$\frac{1}{2}$	50	55
1	0.015	$\frac{1}{2}$	50	41
$1\frac{1}{2}$	0.015	1 or $1\frac{1}{4}$	100 or 125	27
2	0.015	$1\frac{1}{2}$	150	20

[a] Exclusive of core, wrapping and box.

Measurement of Thickness.

13. The thickness shall be measured with a micrometer graduated to 0.001 in., having a circular foot, 0.24 to 0.26 in. in diameter and with a pressure on the specimen of not less than 8 oz. nor more than 10 oz. Five measurements shall be made at random in a length of not less than 3 ft. and no result shall be outside the limits prescribed.

Permissible Variations.

14. The width shall not vary from the specified value by more than $\frac{1}{32}$ in. The thickness shall not vary from the specified value by more than 0.002 in.

PACKING AND MARKING

Packing.

15. Each roll shall be wrapped in paraffined paper or metal foil and enclosed in a suitable box. The wrapping shall be secure and shall thoroughly protect the contents.

Marking.

16. Each box shall be marked with the name of the manufacturer or a trade mark together with the nominal width and weight of the tape.

INSPECTION AND REJECTION

Time of Inspection.

17. The tape shall be inspected and tested within four weeks from the date of delivery.

Retests and Rejections.

18. If the tape fails in any one test of those prescribed in these specifications, two additional specimens will be taken and submitted to that test. If either of these two additional specimens fails, the lot of tape represented by that sample shall be rejected.

STANDARD METHODS OF TESTING RUBBER PRODUCTS[1]

Serial Designation: D 15 – 24

These methods are issued under the fixed designation D 15; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ADOPTED, 1915; REVISED, 1924.

CHEMICAL TESTS

(*A*) *Short Procedure.*

GENERAL INSTRUCTIONS

1. Blank tests shall be run on all determinations and deductions made accordingly. **Blanks.**

2. In the event of any determination not falling within the limits given in these methods of test, a duplicate determination which shall agree within the limits specified shall be made and the average value taken as the true value. **Checks.**

3. (*a*) A specimen weighing not less than 15 g. shall be prepared, taking pieces from various parts of the sample. **Preparation of Specimen.**

(*b*) The specimen shall be cut into small pieces and run through a grinder until all of it will pass through a No. 20 sieve.[2] Care must be taken that the grinder does not become appreciably warm. The backing shall be buffed off before grinding.

4. A strong magnet shall be passed through the specimen to remove any metal from the grinder, and the specimen shall be mixed thoroughly and put in tightly stoppered bottles. It shall not be exposed to sunlight or heat. **Removal of Iron and Care of Specimen.**

REAGENTS

5. Acetone shall be distilled not more than 10 days before use over anhydrous potassium carbonate, using the fraction which distills at 56 to 57° C. **Acetone.**

6. Alcoholic potash shall be of normal strength, made by dissolving the required amount of KOH in absolute alcohol the day before use, and allowing to settle. Only the clear solution shall be used. **Alcoholic Potash.**

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

[2] For detailed specifications for this sieve, see the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials, p. 917.

Barium-Chloride Solution.

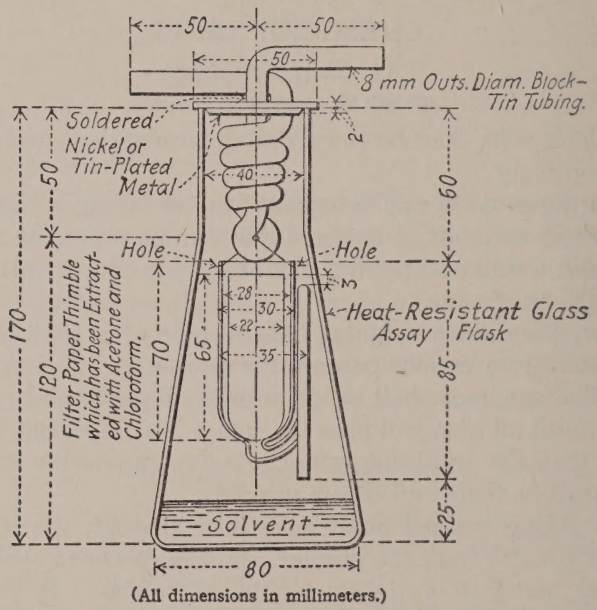
7. Barium-chloride solution shall be made by dissolving 100 g. of crystallized barium chloride in one liter of distilled water and adding two or three drops of concentrated hydrochloric acid. If there is any insoluble matter or cloudiness, the solution shall be heated on the steam bath over night and filtered.

All reagents shall be of a purity equal to that called for in Krauch's "Standard Chemical Reagents; their Purity and Test."

ANALYSES

Acetone Extract.

8. The extraction apparatus shall conform to that shown in Fig. 1. It shall be heated so that the period of filling an empty siphon cup



(All dimensions in millimeters.)

FIG. 1.—Extraction Apparatus.

with acetone and completely emptying it will be between $2\frac{1}{2}$ and $3\frac{1}{2}$ minutes.

Two grams of the rubber shall be extracted continuously with acetone for 8 hours, using a sample that has been prepared within 24 hours. Distill off the acetone and dry the flask and contents for 4 hours at 95 to 100° C. Desiccate until cool and weigh. Continue to dry for 2-hour periods until constant weight is obtained. In drying place the flask on its side but at a sufficient angle from the horizontal so that the extract does not appreciably run down from the side of the flask.

Free Sulfur.

9. Add to the flask containing the acetone extract 50 to 60 cc. of distilled water and 2 or 3 cc. of bromine (if the acetone extract indicates a large amount of free sulfur, the amount of bromine should be increased). Heat gently on the steam bath until the solution is practically colorless and filter into a 400-cc. beaker. Dilute with distilled water until the volume is about 400 cc. Cover the beaker with a watch glass, heat to boiling on the steam bath, add 10 cc. of 10-per-cent barium-chloride solution and allow the precipitate to stand over night. The next day filter off the precipitate, ignite the filter paper and weigh.

Alcoholic-Potash Extract.

10. Dry the residue from the acetone extraction at 50 to 60° C., put into a 200-cc. Erlenmeyer flask with 50 cc. of the alcoholic KOH solution and boil for 4 hours under a reflux condenser. Filter the solution into a beaker and wash twice, using each time 25 cc. of hot absolute alcohol and then wash thoroughly with hot water. Evaporate the solution to approximate dryness, take up in warm water and transfer to a separatory funnel. Acidify with 15 cc. 5 *N* HCl, using this to rinse the beaker. Add sufficient water to make the bulk of the solution 100 cc. When cool, add 40 cc. of ether, using it to rinse the beaker in 20-cc. portions. Shake the aqueous and ethereal solutions thoroughly. After complete separation, draw off the aqueous solution and treat in another separatory funnel, with a fresh 20-cc. portion of ether. Continue to shake the aqueous solution with fresh portions of ether until a colorless portion has been obtained, then shake out twice more. Unite the ethereal solutions and wash with successive additions of water, continuing twice after the water shows no acid reaction. Filter through a plug of extracted cotton into a tared flask, wash the filter and funnel with ether, evaporate the ether without boiling and dry the residue to constant weight at 95 to 100° C. Cool in a desiccator and weigh.

Total Sulfur.

11. Mix a 0.5-g. sample with 4 g. of Na_2O_2 and 6 g. of K_2CO_3 in a dry 15-cc. iron crucible. Cover and heat gradually until the mixture fuses, proceeding cautiously, as rapid heating will cause an explosion. Then bring to quiet fusion for 15 to 20 minutes, applying heat so as to avoid contamination with sulfur fumes. Rotate the crucible while the melt solidifies. When cool, put crucible and cover into a casserole containing 200 cc. of water; add 5 to 10 cc. of bromine water and boil until the melt is dissolved. Allow the precipitate to settle, decant the liquid through a thick filter and wash the residue with hot water. Acidify the filtrate with HCl, evaporate to dryness and dehydrate if silica is present; add 2 cc. of concentrated HCl, take up in water, filter and wash, making the total volume about 400 cc.

Heat to boiling and add slowly a slight excess of hot 10-per-cent barium-chloride solution. Allow to stand over night, filter, wash, ignite, weigh the $BaSO_4$ and calculate to sulfur.

Ash.

12. Weigh out a 1-g. sample in a porcelain crucible, heat in a muffle furnace, the temperature of which is carefully regulated so that no material amount of visible products are given off. After the mass is charred, the temperature shall be raised sufficiently to burn the carbon. The whole process shall be conducted at as low a temperature as possible. At the end of this operation the crucible shall be removed from the furnace, cooled in a desiccator and weighed; the ash broken up and inspected for carbon. If any visible carbon is present, a new determination shall be conducted.

CALCULATIONS

Calculations.

13. The percentage of rubber shall be considered to be the difference between 100 and the sum of the total sulfur and ash expressed as percentages and figured on the total compound. If the alcoholic-potash extract is over 2 per cent of the rubber as first calculated, subtract this excess also from the rubber. The organic-acetone extract shall be obtained by taking the difference between the total acetone extract and the free sulfur. The organic-acetone extract, free sulfur, total sulfur and alcoholic-potash extract shall be figured on the amount of gum as found by the above procedure.

CHECK ANALYSES

Check Analysis.

14. Duplicate determinations when required shall check within the following limits, expressed as percentages of the gum present, as found by analysis, except as stated.

	CHECK WITHIN, PER CENT.
Organic-acetone extract	0.10
Free sulfur	0.10
Total sulfur	0.10
Alcoholic-potash extract	0.10
Ash calculated on total compound	0.25

(*B*) *Complete Procedure.*

NOTE.—The following is the procedure of the Joint Rubber Insulation Committee and was originally prepared for 30-per-cent Hevea rubber insulation for wires and cables. It is applicable to similar rubber compounds used for other purposes, but obviously in such cases the references in this procedure to insulation for wires and cables should be disregarded.

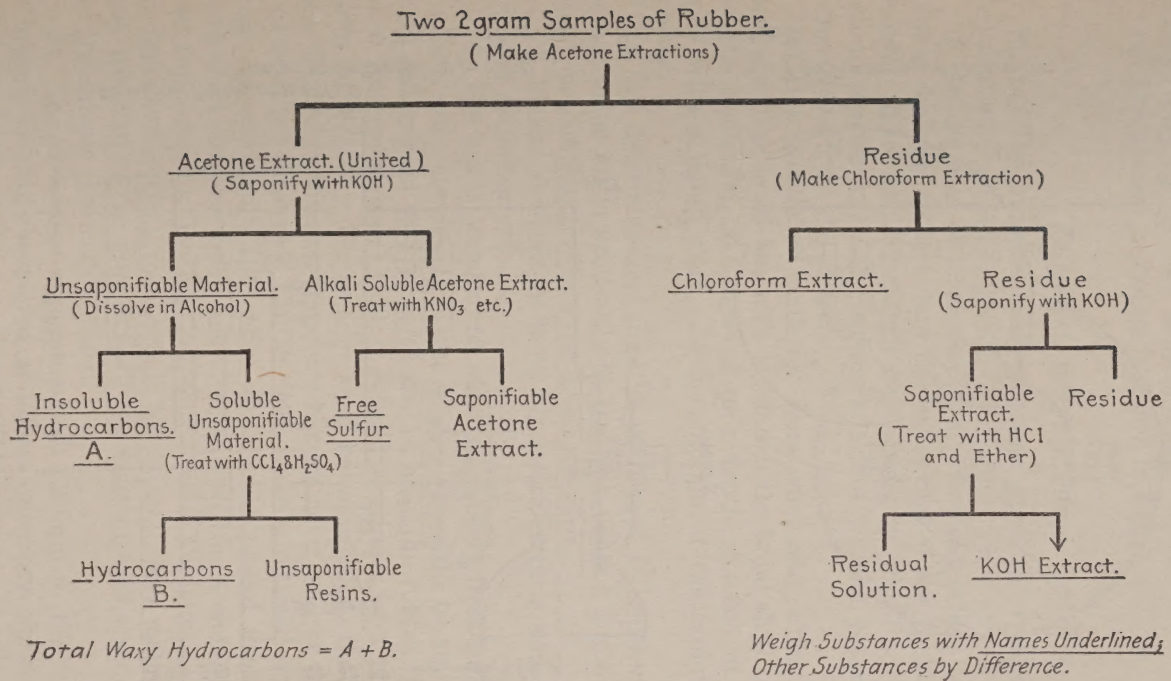


FIG. 2.—Outline of Method For Rubber Analysis.
(Exclusive of Fillers and Total Sulfur Determinations.)

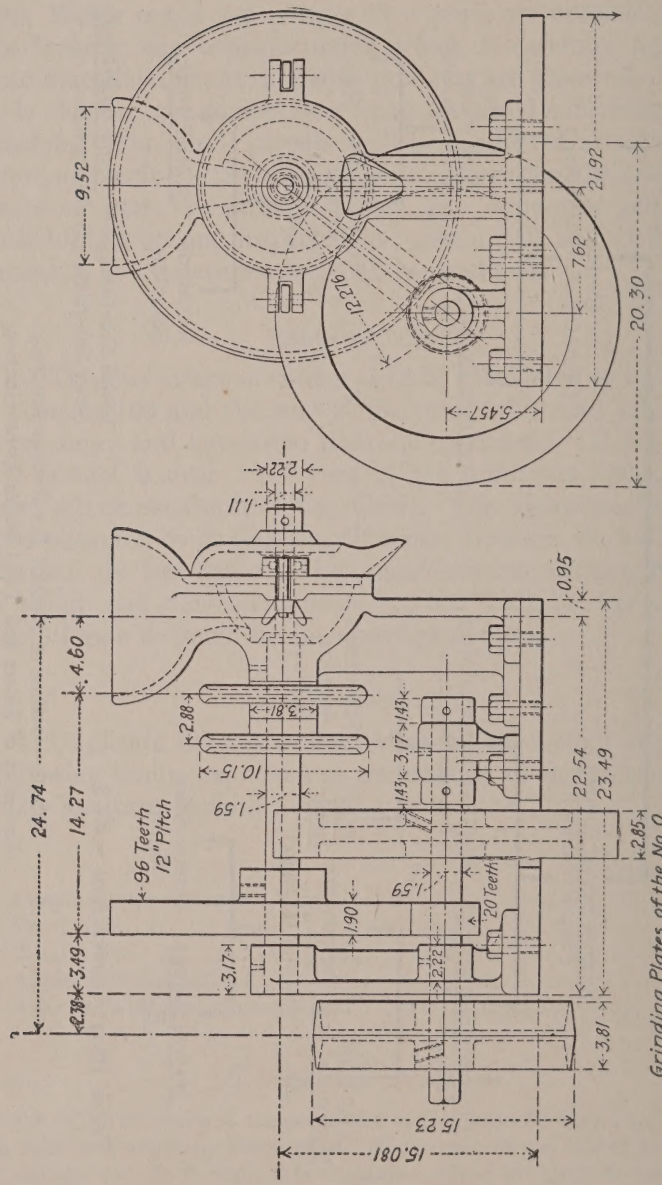


FIG. 3.—Diagram for Rubber Grinder.

15. The object of this procedure of analysis is to determine whether rubber compounds comply chemically with specification which are intended to secure compounds containing 30 per cent of the best Hevea rubber, and mineral fillers. Object of Analysis.

16. The general procedure is shown in Fig. 2. Outline.

GENERAL INSTRUCTIONS

17. (*a*) The analysis shall be made upon the compound after vulcanization. Samples.

(*b*) In the case of insulation from wires and cables, samples for analysis shall be taken before the saturation of the braid whenever possible. The insulation shall be thoroughly wiped with a damp cloth to remove any adhering material but without removing waxy hydrocarbons from the surface.

(*c*) If a sample of insulation from wires and cables has to be taken after the braid has been saturated, the braid shall be removed and the insulation sand-papered to a depth of 0.005 in. and then wiped with a damp cloth. The condition of the sample shall be reported.

18. All determinations shall be performed in duplicate and the average value taken arbitrarily as the true value. Duplicate determinations shall check within the limits specified. Duplicate Analyses.

19. Blanks shall be made on all determinations and the results deducted accordingly. Blanks.

20. The insulation shall be removed entirely from sufficient wire to give a sample weighing about 25 g. This shall be cut into small strips and ground slowly, so as not to heat the rubber, in either a No. 0 Enterprise coffee mill, a Russwinn grinder or a mill such as shown in Fig. 3. The grinder shall be adjusted so that not more than 20 per cent will pass through a No. 40 sieve. All of the material shall be sifted through a No. 20 sieve, what is retained on the sieve being reground until the entire sample has passed through. The sieves shall conform to the requirements of the Standard Specifications for Sieves for Testing Purposes (Serial Designation: E 11) of the American Society for Testing Materials.[1] Any metal that may have come from the grinder shall be removed with a strong magnet and the sample thoroughly mixed. Preparation of Sample of Insulation.

21. The extraction apparatus shall conform to Fig. 1. It shall be heated so that the period of filling an empty siphon cup with acetone and completely emptying it will be between 2½ and 3½ minutes. Extraction Apparatus.

[1] See p. 917.

REAGENTS

Acetone. 22. Acetone shall be freshly distilled over anhydrous K_2CO_3, using the fraction 56–57° C.

Alcoholic Potash. 23. Alcoholic KOH solution shall be of normal strength and shall be made freshly by dissolving the proper amount of KOH (purified by alcohol) in 95-per-cent alcohol which has previously been distilled over KOH. The solution shall be allowed to stand for 24 hours and only the clear liquid used.

Ether. 24. Ether shall be washed with three successive portions of distilled water and distilled, using the fraction 34–36° C.

Chloroform. 25. Chloroform shall be pure and freshly distilled.

Carbon Tetrachloride. 26. Carbon tetrachloride shall be pure and freshly distilled.

27. Reagents not otherwise specified shall be chemically pure. Water shall be distilled.

ANALYSES

Acetone Extract. 28. Extract continuously with 60 cc. of acetone for 8 hours, two 2-g. samples that have been prepared within 24 hours. Unite the extracts in a weighed flask, using hot chloroform to rinse the flasks. Distill off the reagents and dry the flask and contents for 4 hours at 95 to 100° C. Desiccate until cool and weigh. Continue to dry for 2-hour periods until constant weight is obtained. In drying, place the flask on its side but at a sufficient angle from the horizontal so that the extract does not appreciably run down the side of the flask.

Unsaponifiable Matter. 29. Add to the acetone extract 50 cc. of alcoholic KOH solution, boil under a reflux condenser for 2 hours, and evaporate to dryness, removing all alcohol. Add 10 cc. of water and 20 cc. of ether; heat until the wax, etc., are in solution, cool, transfer to a separatory funnel, wash out the flask with warm water and then cool, finally with two 20-cc. portions of ether. The water volume should be 100 cc. and the ether at least 40 cc. Shake vigorously for 2 minutes, and allow the solutions to separate thoroughly. Draw off the aqueous solution into a second funnel, leaving in the first funnel the ethereal solution and any flocculent material that may be present. Again rinse the flask with 20 cc. of ether and add it to the aqueous solution; shake vigorously for 2 minutes, and when separated draw off the aqueous solution and unite in the first funnel the ethereal solutions and any flocculent material. Repeat, shaking with 20-cc. portions of ether until the extraction is complete, using at least 120 cc. of ether. Wash the flask and the funnel, from which the ethereal solution has been taken, with water, until they are free from alkali, subsequently using this wash water to wash the ethereal solution.

Continue washing with water until it has been washed twice after it shows no alkaline reaction. Retain with the ethereal solution any flocculent material. Filter the ethereal solution from the flocculent material, through a small pellet of extracted cotton, into a weighed flask, washing first with ether and subsequently with hot chloroform, using this to rinse the original flask and both separatory funnels. Evaporate the solvents and dry the extract to constant weight at 95 to 100° C.; desiccate until cool and weigh.

Hydrocarbons A.

30. Add 50 cc. of absolute alcohol to the unsaponifiable material and warm until solution is as complete as possible. Cool the solution to −4 or −5° C. and maintain at this temperature for 1 hour by packing the flask in a mixture of ice and salt. Filter out the waxy hydrocarbons, using a funnel packed with ice and salt, and apply suction if necessary. Wash the flask and filter with about 25 cc. of 95-per-cent alcohol, which has been previously cooled in the same temperature. Catch the filtrate in a flask which is afterwards cooled to −4 or −5° C. to make sure that all possible waxy hydrocarbons have been removed, and refilter if necessary. Dissolve the residue on the filter paper with hot chloroform, into the original flask. Evaporate the chloroform and dry the flask to constant weight at 95 to 100° C. Cool in a desiccator and weigh.

Hydrocarbons B.

31. Evaporate the alcohol from the flask containing the alcoholic soluble unsaponifiable material, add 25 cc. carbon tetrachloride and transfer to a separatory funnel. Shake with concentrated H_2SO_4, drain off the discolored acid and repeat with fresh portions of acid until there is no longer any discoloration. After drawing off all the acid, wash the carbon tetrachloride solution with repeated portions of water until all traces of acid are removed. Transfer the carbon tetrachloride solution to a weighed flask; evaporate off the solvent and dry the flask to constant weight at 95 to 100° C. Cool in a desiccator and weigh.

Free Sulfur.

32. Add 2 g. KNO_3 to the aqueous solution and washings from the ethereal separation of the unsaponified material. Evaporate to dryness in a silver or nickel dish and heat to quiet fusion, avoiding contamination with sulfur fumes. Transfer with water to an evaporating dish, acidify with HCl, evaporate to dryness, and dehydrate silica. Add 2 cc. of concentrated HCl, take up in water, filter and wash, making a volume of 200 cc. Heat to boiling and add slowly a slight excess of hot 10-per-cent $BaCl_2$ solution. Allow to stand over night, filter, wash, ignite, weigh the $BaSO_4$ and calculate the sulfur.

Chloroform Extract.

33. Extract continuously the residues from both of the acetone extractions (without necessarily removing the acetone that may be

on them) for 4 hours with 60 cc. of chloroform, unite the extractions in a weighed flask, using hot chloroform to rinse the flasks. Distill off the solvent and dry the flask and contents for 2 hours at 95 to 100° C. Desiccate until cool and weigh. Continue to dry for 1-hour periods until constant weight is obtained. In drying, place the flask on its side but at a sufficient angle from the horizontal so that the extract does not appreciably run down the side of the flask. (If it is needful to wait after the acetone extraction, before starting the chloroform extraction, the sample must be kept in a vacuum of at least 50 mm. of mercury.)

Alcoholic-Potash Extract.

34. Dry the residue from the chloroform extraction at 50 to 60° C. until odor of chloroform can no longer be detected, unite residues from the two 2-g. samples in a 200-cc. Erlenmeyer flask. Add 100 cc. of alcoholic KOH solution and boil for 4 hours under a reflux condenser. Filter the solution by decantation through an 11-cc. hardened filter paper into a beaker and wash twice, using each time 25 cc. of hot absolute alcohol and then wash thoroughly with hot water. Wash any rubber on the filter paper back into the original flask and reserve this for the determination of the rubber hydrocarbons. Evaporate the solution to approximate dryness, take up in warm water and transfer to a separatory funnel. Acidify with 15 cc. of 5 *N* HCl, using this to rinse the beaker. Add sufficient water to make the bulk of the solution 100 cc. When cool, add 40 cc. of ether, using it to rinse the beaker in 20-cc. portions. Shake the aqueous and ethereal solutions thoroughly. After complete separation, draw off the aqueous solution and treat in another separatory funnel, with a fresh 20-cc. portions of ether. Continue to shake the aqueous solution with fresh portions of ether until a colorless portion has been obtained, then shake out twice more. Unite the ethereal solutions and wash with successive additions of water, continuing twice after the water shows no acid reaction. Filter through a plug of extracted cotton into a tared flask, wash the filter and funnel with ether, evaporate the ether without boiling and dry the residue to constant weight at 95 to 100° C. Cool in a desiccator and weigh.

Rubber Hydrocarbons.

35. Add to the flask containing the rubber residue from the alcoholic KOH extraction, sufficient water to make the total volume of the solution 125 cc. and then add 25 cc. of concentrated HCl. Heat for an hour at 97 to 100° C. Decant the supernatant liquid through a hardened filter paper on a Büchner funnel 7 cm. in diameter, using suction; wash the residue with 25 cc. of hot water and decant. (While a Büchner funnel is recommended, it is permissible to use an 11-cm. hardened filter paper with platinum cone, in a 60-deg. funnel.)

Perform this entire treatment with water and HCl three times and save the first and second decantations for the "organic matter" test described in Section 40. The rubber at this stage should be white and practically free from black specks of undissolved fillers; if not, continue the acid treatment until the black specks disappear. (If carbon is present, all the particles of rubber will be grayish, bluish, or black, depending on the form and quantity of carbon used. Black specks in light particles of rubber usually indicate the presence of lead sulfide which must be removed to prevent the formation of lead sulfate on igniting the residue *C*.) Add 150 cc. of hot water to the flask and let stand on a steam bath or hot plate for half an hour and decant through the filter paper, repeating until washings are free from chlorides. (See Section 40.) Transfer all the rubber in the flask to the filter paper and dry as much as possible by suction. Wash the rubber with 50 cc. of 95-per-cent alcohol, using suction. Transfer the entire residue to a weighing bottle. Dry at 95 to 100° C. for an hour, cool in a vacuum desiccator under reduced pressure and weigh. Dry for 30 minutes, cool and weigh, repeating this process until either constant weight is reached or the weight starts to increase. Let this weight be represented by *C*. Determine the ash (*E*) on a portion (*D*) of this residue (*C*) and sulfur (*H*) on the remaining portion (*G*). Also determine the sulfur (*F*) on the ash (*E*). Perform the ash determination as described under ash in Rubber Residue and the sulfur determinations as described under Total Sulfur. Then,

$$\text{Rubber hydrocarbons} = 100\,\frac{C}{4}\left(1-\frac{H}{C}-\frac{E-F}{D}\right)$$

expressed as a percentage of the total sample.

Ash in Rubber Residue.

36. Place about 0.5 g. (*D*) of residue *C* into a weighed porcelain crucible and heat gently, gradually driving off the volatile matter. When the crucible has ceased to smoke, raise the temperature gradually to between 450 and 500° C., until all organic matter has been burned away, which is usually indicated by the ash becoming white. (An electric muffle furnace with pyrometer is recommended for this purpose.) Cool in a desiccator and weigh, the weight of ash being represented by *E* in the formula for rubber hydrocarbons. Make the sulfur test on ash by the method described in Section 37. If, however, the ash (*E*) is not over 0.08 *D*, the determination of sulfur in the ash may be omitted, and *F* assumed to be zero.

Total Sulfur

37. Mix a 0.5-g. sample with 4 g. of Na_2O_2 and 6 g. of K_2CO_3 in a dry 15-cc. iron crucible. Cover and heat gradually until the mix-

ture fuses, proceeding cautiously, as rapid heating will cause an explosion, and then bring to quiet fusion for 15 to 20 minutes. Apply the heat so as to avoid contamination with sulfur fumes. Rotate the crucible while the melt solidifies. When cool, put the crucible and cover into a casserole containing 200 cc. of water; add 5 to 10 cc. of bromine water and boil until the melt is dissolved. Allow the precipitate to settle, decant the liquid through a thick filter and wash the residue with hot water. Acidify the filtrate with HCl, evaporate to dryness and dehydrate silica; add 2 cc. of concentrated HCl, take up in water, filter and wash, making the total volume about 100 cc. Heat to boiling and add slowly a slight excess of hot 10-per-cent $BaCl_2$ solution. Allow to stand over night, filter, wash, ignite, weigh the $BaSO_4$ and calculate to sulfur.

Specific Gravity.

38. The specific gravity shall be the ratio of the weight of a given volume of the rubber to the weight of an equal volume of water, both at 20° C. Strips of the largest applicable size shall be cut from the conductor and about 5 g. used for the sample. The specific gravity shall be determined in the usual manner by means of a specific gravity bottle. Care must be taken that no air bubbles adhere to the sample.

Red Lead.

39. Dissolve 1 g. of the sample in 75 cc. of xylol at a temperature of about 100° C. When the rubber is dissolved, the absence of any red particles indicates the absence of red lead. If red particles are present, filter the solution into a Gooch crucible and wash thoroughly with benzol, acetone and alcohol successively. Remove the felt and residue to a distilling flask, add HCl, and distill over the chlorine liberated by the lead peroxide, absorbing the gas in a solution of KI and starch. Not more than 0.1 cc. of 0.1 *N* thiosulfate shall be required to titrate the iodine liberated.

Organic Filler.

40. Transfer the first and second decantations of the HCl solutions to a carefully cleaned porcleain dish and add 20 cc. of concentrated H_2SO_4. Place the dish on a steam bath or hot plate to drive off water and HCl. A pronounced charring of the residue indicates the presence of organic matter soluble in water or hydrolyzed by HCl. Examine the filter paper and rubber while decanting the acid solution and washing free of chlorides. Some types of organic fillers not removed by water and HCl would be plainly visible at this point. Place a small portion of residue *C* under a microscope and examine for fibrous and other characteristic organic material. If organic fillers are indicated and not clearly proved by this test, place 1 g. of the original sample in a beaker, add 50 cc. of xylol and heat on a hot plate until the rubber is dissolved. Decant the xylol solution and wash

the residue with ether several times by decantation. Dry the residue and examine under the microscope.

DEFINITIONS OF TERMS DESCRIBING COMPONENTS OF ACETONE EXTRACT

41. *Organic Acetone Extract.*—In the case of chemical analysis of rubber, the difference between the acetone extract and the free sulfur. Organic Extract.

42. *Saponifiable Acetone Extract.*—In the case of chemical analysis of rubber, the difference between the organic acetone extract and the unsaponifiable material. Saponifiable Acetone Extract.

43. *Total Waxy Hydrocarbons.*—In the case of chemical analysis of rubber, the sum of the hydrocarbons A and B. Waxy Hydrocarbon.

44. *Unsaponifiable Resins.*—In the case of chemical analysis of rubber, the difference between the unsaponifiable material and the waxy hydrocarbons. Unsaponifiable Resins.

CHECK ANALYSES

45. Specific gravity determinations shall check within 0.01. The other duplicate determinations shall check within the following limits expressed as percentages of the original sample. Checks.

DETERMINATION.	CHECK.
Acetone extract	0.10
Saponifiable acetone extract	0.10
Unsaponifiable resins	0.10
Waxy hydrocarbons	0.10
Free sulfur	0.05
Chloroform extract	0.10
Alcoholic potash extract	0.10
Rubber hydrocarbons	0.20
Total sulfur	0.10

INTERPRETATION

46. The rubber shall be considered to be the sum of the rubber hydrocarbons, saponifiable acetone extract, unsaponifiable resins and chloroform and alcoholic potash extracts, expressed as percentages. If the chloroform extract is over 3.0 per cent of the rubber so calculated, subtract the excess from the rubber. If the KOH extract is over 1.8 per cent of the rubber as first calculated, subtract this excess also from the rubber. Interpretation.

STATEMENT OF RESULTS

47. The results of the analysis shall be stated in the following form: Statement of Results.

	PER CENT.
Acetone extract	-------
Saponifiable acetone extract	-------
Unsaponifiable resins	-------
Waxy hydrocarbons	-------
Free sulfur	-------
Chloroform extract	-------
Alcoholic potash extract	-------
Total sulfur	-------
Rubber	-------
Color of acetone extract (60 cc. vol.)	-------
Fluorescence in acetone extract solution (present or absent)	-------
Hydrocarbons A (consistency and color	-------
Hydrocarbons B (solid or liquid)	-------
Color of chloroform extract (60 cc. vol.)	-------
Carbon (present or absent)	-------
Red lead (present or absent)	-------
Specific gravity	------

PHYSICAL TESTS

Scope.

48. (*a*) These tests are intended to apply to soft vulcanized rubber compounds such as are used for lining and cover of hose, belting, gloves used by electrical workers, etc.

(*b*) Only those tests which are applicable to rubber products in general have been covered. Tests which are special for only one class of material are not included since such tests more properly belong in the specifications for the particular material.

Selection and Condition of Samples.

49. (*a*) Samples shall be selected in accordance with the requirements of the specifications covering the material to be tested.

(*b*) The samples shall be taken from the finished product and shall not be heated, immersed in water or subjected to any mechanical or chemical treatment not specifically prescribed herein.

Age of Samples.

50. No tests shall be made within 48 hours after vulcanization unless agreed to by the manufacturer.

Temperature.

51. (*a*) The temperature of the surrounding air where samples of rubber are being tested shall not be less than 65° F. (18.3° C.) nor more than 90° F. (32.2° C.).

(*b*) The samples shall be kept at a temperature within these limits for at least 3 hours immediately before being tested.

Separation of Rubber.

52. When the tests to be made require the separation of the rubber from cotton fabric, it shall be done without the use of gasoline if possible. If it is necessary to use gasoline, it shall be of a grade which distills below 239° F. (115° C.) and the specimen shall be allowed to rest at least one hour before being tested.

(*A*) *Tensile Strength, Elongation and Set Tests.*

TENSION TESTING APPARATUS

53. (*a*) Tensile strength and elongation tests shall be made on a power-driven apparatus, preferably of the inclination-balance or pendulum type. A spring balance type of apparatus may be used if provided with a device which will indicate the actual maximum load at which rupture took place, and provision is made to prevent recoil of the spring. Type.

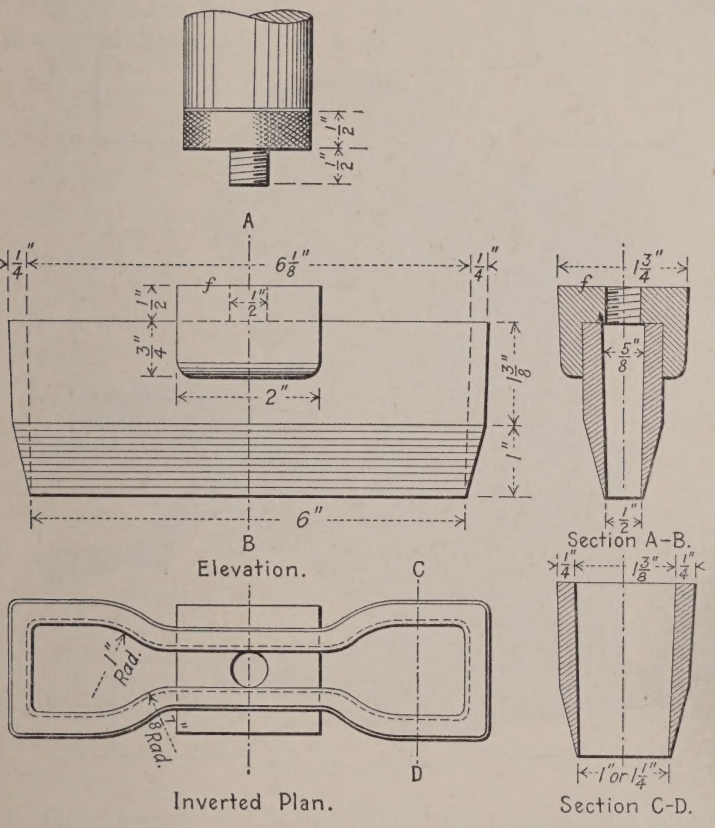


FIG. 4.—Die for ½ by 2-in. Specimens.

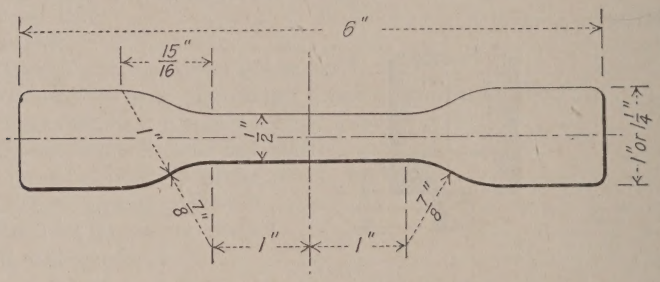
(*b*) The accuracy of the apparatus shall be such that the readings obtained are correct within ±1 per cent. Accuracy.

(*c*) The grips which hold the specimen in the testing machine shall be of any desired type which tightens automatically and exerts a uniform pressure across the width of the specimen proportional to the applied tension. Grips.

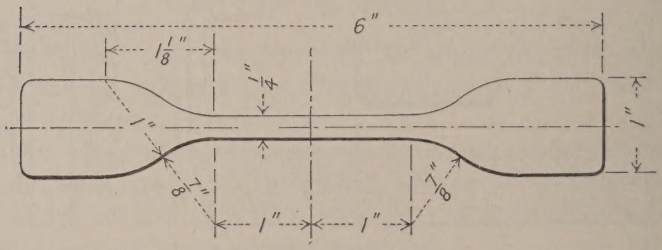
Rate. (*d*) The rate of separation of the grips shall be uniform at all times and approximately 20 in. per min.

TEST SPECIMENS

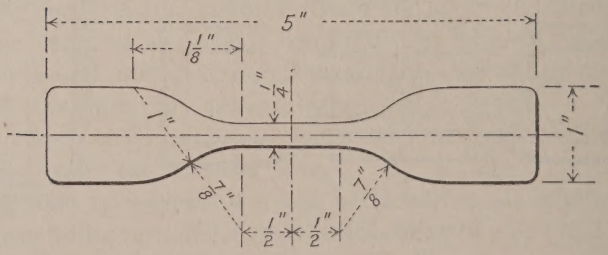
Shape. 54. (*a*) Unless otherwise prescribed in the specifications for the material being tested, the specimens for tensile strength and elongation tests and those for set tests shall be stamped out with a steel die



(*a*) ½ by 2-in. Specimen.



(*b*) ¼ by 2-in. Specimen.



(*c*) ¼ by 1-in. Specimen.

FIG. 5.—Tensile Strength and Elongation Test Specimens.

constructed substantially in accordance with Fig. 4, the specimen to have a reduced section at the center 2 in. long and 0.5 in. wide, 2 in. long and ¼ in. wide or 1 in. long and ¼ in. wide as shown in Fig. 5. The largest specimen that can be readily cut from the sample shall be used.

(*b*) All backing and deep corrugations as in seamless woven or braided hose shall be carefully removed by means of a suitable grinding machine and without causing excessive heating. It is recommended that a grinder similar to that shown in Fig. 6 be used. **Buffing.**

At least one hour shall elapse between the buffing and testing of the specimen.

(*c*) Two parallel lines perpendicular to the axis of the specimen shall be marked on the reduced section, one on each side of the center and 0.5 in. or 1 in. therefrom as required. The marks shall be as narrow as possible consistent with legibility. **Gage Marks.**

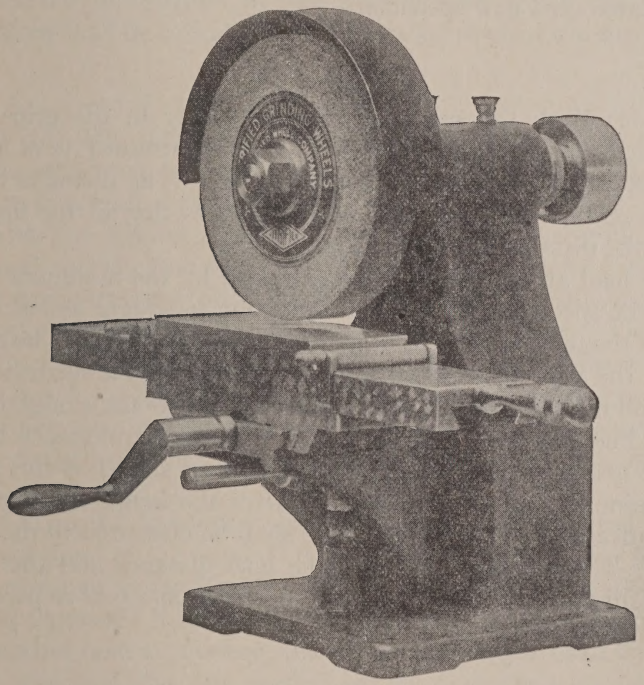


FIG. 6.—Grinder.

(*d*) The thickness shall be measured with a micrometer graduated to 0.001 in. having circular shoes 0.24 to 0.26 in. in diameter and with a pressure on the specimen of not less than 8 oz. nor more than 10 oz. **Dimensions.**

The minimum thickness and width between the gage marks shall be taken in calculating the area to be used in determining the tensile strength of the specimen.

55. (*a*) One specimen per sample shall be tested for tensile strength and elongation. If either the tensile strength or the elongation is below the specified value, two additional specimens shall be **Number of Test Specimens.**

tested. If either specimen falls below the specified value for either tensile strength or elongation, the material represented by the sample from which the specimens were taken shall be considered to have failed to meet the specifications.

(*b*) One specimen per sample shall be tested for set. If it fails to meet the specifications, the procedure shall be the same as in Section 55 (*a*).

(*c*) If a specimen breaks outside of the gage marks in the tensile strength and elongation tests, or is found to have been defective in either the tension test or the set test, it shall be discarded and the test repeated on a new specimen.

PROCEDURE

Tensile Strength and Elongation.

56. (*a*) With the specimen gripped evenly in the grips of the testing machine, the grips are separated at a uniform rate of approximately 20 in. per min. until rupture occurs. The distance between the gage marks shall be noted continuously so that at the instant of rupture the distance is known within 0.1 in.

The load at rupture in pounds divided by the minimum original area of the specimen at the reduced section in square inches shall be taken as the tensile strength in pounds per square inch. The distance between the gage marks at rupture in inches to the nearest 0.1 in., minus the original gage length, shall be taken as the elongation.

Set.

(*b*) The specimen shall be stretched until the distance between the gage marks is the prescribed amount, held stretched this amount for 10 minutes and then released. After the lapse of 10 minutes, the distance between the gage marks shall be measured to the nearest 0.02 in. The difference between the final distance and the original distance, divided by the original distance and expressed as percentage, shall be taken as the set.

(*B*) *Friction Tests.*

TEST SPECIMENS

Fire Hose.

57. (*a*) The test specimens shall be pieces of the hose 2 in. in length accurately cut from the sample with a sharp tool which will leave clean edges. These rings shall then be slit in the lengthwise direction of the hose and laid out flat. One-quarter inch of the rubber lining shall be carefully and cleanly trimmed off on each side without injuring the fabric leaving a strip of rubber lining $1\frac{1}{2}$ in. in width undisturbed on a strip of cover 2 in. in width.

(*b*) The test specimens shall be pieces of the hose 1 in. in length accurately cut from the sample with a sharp tool which will leave clean edges. They shall be tested in the ring form. Other Types of Hose.

(*c*) A strip 6 in. in length by 1 in. in width shall be cut from the sample of belting with a sharp tool which will leave clean edges. Two test specimens of two plies each shall be taken from this strip and tested separately. Belting.

58. One specimen per sample shall be tested for friction. If the result is below the specified value, two additional specimens shall be tested. If either of these specimens falls below the specified value, the material represented by the sample from which the specimens were taken shall be considered to have failed to meet the specifications. Number of Test Specimens.

PROCEDURE

59. (*a*) The plies, the friction between which is to be tested, shall be separated by hand at one end of the specimen a sufficient distance to permit attaching the grips. Fire Hose and Belting.

With one separated end clamped in a stationary grip, the other separated end is clamped in a freely suspended grip hanging vertical and to which the prescribed weight is attached with suitable provision for supporting and releasing it slowly without jerking. The distance through which separation takes place shall be noted for a period of 10 minutes if possible. The friction shall be taken as the rate obtained by dividing the total distance separated in inches (to nearest 0.05 in.) by the elapsed time in minutes (to the nearest 0.017 min., that is, one second.)

(*b*) The test specimen shall be placed on a mandrel having a diameter equal to the nominal diameter of the hose. The cover shall be cut through in the lengthwise direction of the hose and enough of one end separated from the rubber to permit attaching a grip. With the mandrel held stationary, the prescribed weight shall be attached to the grip and the test carried out as prescribed for fire hose in Paragraph (*a*). Other Types of Hose.

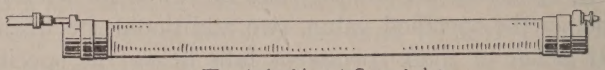
(*C*) *Hydrostatic Tests of Hose.*

60. Pressure tests shall be made with water by means of a pump or by means of an accumulator system. The pressure shall be increased at a constant rate of 300 lb. per min. and shall be measured with a calibrated gage. Application and Measurement of Pressure.

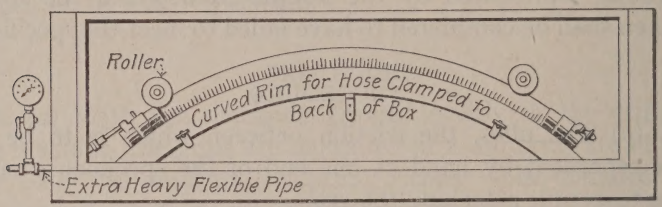
61. (*a*) *Tests of Each Length.*—The hose shall be stretched on a plane surface in a straight line and connected to the water line or pump and filled with water, the air being allowed to escape through Fire Hose.

an air cock. When the hose is filled with water the air cock shall be closed, a pressure of 10 lb. per sq. in. applied and the length carefully measured.

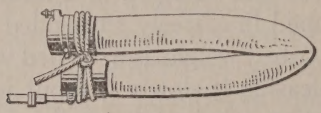
The pressure shall then be increased to the prescribed value and held for the prescribed period. While still under this pressure the length shall be measured and the amount of twist, warp and rise shall be determined.



Test 1. Hose Straight.



Test 2. Hose Curved on Radius of $2\frac{1}{4}$ feet.



Test 3. Hose Kinked, Ends Tied Together.

FIG. 7.—Apparatus for Pressure Test.

(*b*) *Tests of Samples.*—The arrangement of the sample for various proof tests—straight, curved and kinked—shall be substantially as indicated in Fig. 7 except that in the kink test of a full length, the two parts of the hose made by the kink shall be tied together at a point 18 in. from the kink.

If the sample fails in this test, two additional samples shall be tested and if either of these fails, the entire lot of hose represented by the samples shall be considered to have failed to meet the specifications.

Other Types of Hose.

62. In testing samples of other types of hose, the pressure shall be raised at the above rate until the prescribed pressure is reached and then held for the prescribed period, after which the pressure shall be increased at the same rate until failure occurs.

If the sample fails in this test, two additional samples shall be tested and if either of these fails the entire lot of hose represented by the samples shall be considered to have failed to meet the specifications.

STANDARD SPECIFICATIONS

FOR

TEXTILE TESTING MACHINES[1]

Serial Designation: D 76 – 27

These specifications are issued under the fixed designation D 76; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1920; ADOPTED IN AMENDED FORM, 1927.

Tensile Testing Machines

Type of Machine.

1. Textile testing machines shall be of the inclination balance or pendulum type.

Angle of Pendulum.

2. The maximum angle of swing of the pendulum in textile testing machines shall be 45 deg. from the vertical.

Pendulum Drum.

3. The minimum diameter of drum for transferring the pull on the specimen to the swinging pendulum shall be 2 in.

Testing Machine Capacity.

4. In selecting the proper capacity of a textile testing machine for a given sample of fabric or yarn, the maximum capacity of the machine shall not exceed that at which the pendulum reaches a swing of 45 deg. from the vertical. The minimum capacity of the machine when used for a given sample of fabric or yarn shall not be less than 20 per cent of the above maximum capacity.

Type of Jaw.

5. (*a*) *Fabric Jaws.*—The clamps of textile testing machines for use upon fabrics shall consist of flat metallic jaws pressing directly against the specimen. One gripping surface shall be hinged or swiveled and the other shall be rigidly connected to the frame of the jaw. The pressure between the jaws shall be secured by any suitable mechanical device so constructed as to grip the fabric firmly before the testing load is applied and to prevent visible slippage during the progress of the test.

(*b*) *Skein Jaws.*—The drums of testing machines for yarn skeins shall consist of cylindrical spools not less than 1 in. in diameter and not less than 1 in. in width, so supported that at least one shall turn freely upon its axis.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

(*c*) *Individual Strand Jaws.*—The jaws or clamps for tests upon individual strands of yarn shall be of the cylindrical or drum type, so arranged that the strands of yarn shall pass around not less than 180 deg. circumference before being clamped or fixed in the jaw. The length of the specimen shall be considered from center to center of drums. The minimum diameter of the cylinder or drum shall be $\frac{1}{2}$ in.

Jaw Dimensions.

6. The width of jaw in a direction perpendicular to the specimen shall in no case be less than 1 in. The depth of jaw in a direction length-wise of the specimen shall in no case be less than 1 in.

Backlash.

7. The dial pointer of textile testing machines shall be so arranged as to be easily adjustable to a zero reading for any weight of jaw or other fixture in the testing machine. The dial pointer shall be so counter-weighted as to prevent undue fluctuations in its position due to backlash, whatever the dial reading may be.

Machine Speed.

8. Textile testing machines shall be power driven or operated in such a manner as to produce a uniform and accurate movement of 12 in. per minute for the pulling jaw.

Calibration.

9. In calibrating textile testing machines, dead weights of accurate amounts shall be used, but these weights shall be applied at a speed of 12 in. per minute, corresponding to the standard jaw speed. The machine shall otherwise be arranged in an entirely similar manner to that used in testing fabric.

STANDARD SPECIFICATIONS

FOR

TOLERANCES AND TEST METHODS FOR COTTON YARNS, SINGLE AND PLIED[1]

Serial Designation: D 180 – 27

These specifications are issued under the fixed designation D 180; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED IN AMENDED FORM, 1927.

Scope.

1. These specifications cover the tolerances and methods of testing for cotton yarn, both single and plied.

TOLERANCES

STRENGTH

Strength of Yarn.

2. The average tensile strength of each case, bale, chain ball or beam warp of yarn, either single or plied, as found by test shall be not less than the specified strength.

SIZE OR YARN NUMBER

Size of Yarn, Carded and Combed.

3. The average size of each case, bale, chain ball or beam warp of yarn, in the singles, either carded or combed, as found by test shall not vary more than 3 per cent above or below the specified size.

Example.—No. 36 carded yarn specified; tolerance would allow 34.92 to 37.08.

TWIST

Direction of Twist.

4. *Direction of Twist.*—The direction of twist shall be as defined in the Standard Definitions of Terms Relating to Textile Materials (Serial Designation: D 123) of the American Society for Testing Materials, as follows:

Twist, Direction of.—In the case of yarn or cord, the yarn or cord has right-hand or regular twist if when it is held vertically the spirals or twists are seen to incline upward in a right-hand direction

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

and has left-hand or reverse twist when the spirals or twists are seen to incline upward in a left-hand direction.

NOTE.—Attention is called to the fact that this definition of twist is opposite to that used in sewing thread manufacture.

Twist of Plied Yarns.

5. The average twist of each case, bale, chain ball or beam warp of plied yarns as found by test shall not vary more than 5 per cent above or below that specified.

Example.—Twist specified is 20 twists per inch; tolerance 19 to 21.

METHODS OF TESTING

STRENGTH

Two test methods are given, the skein test for single yarn and the single strand test for single and plied yarns. A Preferred and Alternative Method for each test is given. The Alternative Method can be used where routine testing is done on a large scale. The Preferred Method should always be used in case of dispute.

Strength, Single Yarn, Skein Preferred.

6. *Skein Test (Preferred Method).*—A standard skein (120 yd.) shall be broken after conditioning of tubes or bobbins selected for test for 12 hours or of skeins for at least 3 hours in an atmosphere of 65 per cent relative humidity, 70° F. (21° C.). An automatic power yarn tester of inclination balance type, the maximum capacity of which shall be determined in accordance with a table of machine specifications, shall be used. The drums of the testing machine shall consist of cylindrical spools not less than one inch in diameter and not less than one inch in width, so supported that at least one shall turn freely upon its axis. The speed of the pulling jaw shall be 12 in. per minute. Any yarn reel having a 1½-yd. perimeter may be used in preparing the skeins. For filling-wound yarns or yarns on cones, where the yarn is drawn from the top, a speed of 100 to 300 r.p.m. of reel shall be used. For warp-wound yarns or yarn on parallel tubes where the yarn is drawn from the side, a speed of 20 to 30 r.p.m. of reel shall be used. On reels that have only one pigtail guide, the tension shall be applied by making one full wrap of the yarn around the guide. On reels using two or more guides, the yarn shall pass straight through the guides onto the reel, the angles of the guides supplying the necessary tension. Judgment must be used in regard to the amount of tension required on yarns having little or a large amount of twist. Three tests from each of four bobbins from every case of yarn shall be made.

7. *Strength Yarn on Beams (Skein Test for Yarn on Beams).*—Yarn received on beams shall be given the skein test and skeins shall be prepared in the following manner: Place the beam containing the yarn to be tested on two bearings sufficiently high from the floor so

that the beam can be turned easily. Directly in front of the beam and at least 15 ft. from the beam, a small table shall be placed, the top of which will be approximately the same height as the top of the beam of yarn. On this table a standard yarn reel shall be placed directly in front of the beam. A small crank arm shall be attached to one end of the beam shaft. Four ends of yarn, taken from the beam, shall be attached to the yarn reel in the regular manner, such as is done when testing yarn from bobbins. One operative shall turn the beam slowly in the proper direction so that the yarn will unwind, and the second operative shall turn the reel fast enough to take up the yarn as it comes off the beam. This operation shall be continued until 120 yd. or the desired length for testing has been taken from the beam. The four skeins shall then be taken from the reel and tested in the regular manner. If it should be necessary to test more ends, the same method of testing shall be followed.

NOTE.—In using these beams after these ends have been taken off, a set of spools containing the same kind of yarn as is on the beam can be placed in behind the beam on a small creel, to fill out the number of ends that have been used for testing. When the ends come up on the beams that have been used for testing, the auxiliary spools in back of the beams can be broken out.

Strength, Single Yarn, Single Strand Preferred.

8. *Single Strand Test (Preferred Method).*—Single strands shall be broken after conditioning the tubes or bobbins for 12 hours in an atmosphere of 65 per cent relative humidity, 70° F. (21° C.). A single strand tester of proper capacity with the jaws set 10 in. between grips and having a speed of pulling jaw of 12 in. per minute shall be used. The average of 4 breaks from each of 10 bobbins shall be the average strength.

Strength, Plied Yarns, Single Strand Preferred.

9. *Plied Yarns (Preferred Method).*—Plied yarns, except standard tire cord, shall be subjected to the single strand break after conditioning for 12 hours on spools or tubes selected for test, in an atmosphere of 65 per cent relative humidity and 70° F. (21° C.). Standard tire cord shall be tested under dry conditions in accordance with the Standard General Methods of Testing Woven Textile Fabrics (Serial Designation: D 39) of the American Society for Testing Materials.[1] A single strand tester of proper capacity with the jaws set 10 in. between grips and having a speed of pulling jaw of 12 in. per minute shall be used. The average of 4 breaks from each of 10 spools or tubes shall be reported as the average strength.

Strength for All Yarns, Alternative Method.

10. *Skein and Single Strand (Alternate Method).*—Skeins or single strands of yarn either single or plied, prepared in accordance with Sections 6, 7, 8 and 9, shall be broken under natural humidity condi-

[1] See p. 900.

tions at time of test. The results thus obtained shall be reduced to a common basis of standard moisture regain equal to 7 per cent of the bone-dry weight.

Moisture Regain Determination.

11. To determine moisture regain present in samples, the several skeins shall be weighed collectively, immediately after testing, under natural moisture conditions which obtain at the time of test. The skeins shall then be placed in the basket of an oven at a temperature of 105 to 110° C. (221 to 230° F.) and dried to constant weight. The moisture regain is then computed as the percentage of the dry weight.

Correction to Standard Regain.

12. (*a*) The following formula shall then be applied, based on the assumption that the standard moisture regain of cotton yarns is 7 per cent of the dry weight, that the actual percentage regain is between the limits of 3 and 7 per cent of the dry weight and that for 1 per cent of moisture regain there is an increase of 6 per cent in the tensile strength of the yarn.

$$\text{Tensile strength corrected to standard moisture regain} = \frac{\text{(Tensile strength from machine reading)} \times 142}{100 + (6 \times \text{actual percentage regain.})}$$

Example.—With a skein of yarn broken under natural conditions showing a tensile strength of 120 lb. as read from the machine dial, and which by weighing before and after drying the skein was found to contain a moisture regain equal to 5 per cent of the bone-dry weight, the tensile strength corrected to a common basis of 7 per cent moisture regain would be:

$$\text{Corrected tensile strength} = \frac{120 \times 142}{100 + (6 \times 5)} = 131 \text{ lb.}$$

(*b*) Moisture regain tests shall be made periodically during the hours of testing as the natural humidity conditions are found to vary.

Strength Correction to Size.

13. The average tensile strength shall be corrected to the specified size as determined in accordance with Sections 14, 15 and 16, by the following formula:

$$\text{Corrected tensile strength} = \text{Actual average strength} \times \frac{\text{Actual average size}}{\text{Specified size}}$$

SIZE OR YARN NUMBER

Size of Single Yarns, Preferred Method.

14. The size of all standard skeins used in the skein strength test described in Section 6 shall be determined immediately after being broken. In case the single strand test is made, the standard skein shall be prepared for the size determination at the time of the break and the size determined immediately. The size of 60-yd. bundles prepared from beams as described in Section 7 shall be determined as soon as possible after conditioning and one test from each beam shall be reported as the average size. The balance to be used in this test shall be accurate to 0.25 per cent of the standard size of the yarn.

When the balance does not indicate the size directly, the yarn number or size may be calculated from the formula:

$$\text{Yarn number or size} = \frac{\text{Length in yards of single yarn}}{\text{Weight in grains}} \times \frac{\text{7000 (grains in 1 lb.)}}{\text{840 (yards of No. 1 cotton yarn per pound)}}$$

Size of Plied Yarns, Preferred Method.

15. In determining the size of plied yarns, the skein shall be prepared in accordance with Table I, and the size shall be determined after conditioning of tubes or spools selected for test for 12 hours or of skeins for at least 3 hours, in an atmosphere of 65 per cent relative humidity, and 70° F. (21° C.). Any yarn reel having a $1\frac{1}{2}$-yd. perimeter may be used in preparing the skeins. For filling-wound yarns or yarn on cones, a speed of 100 to 300 r.p.m. of reel shall be used. For warp-wound yarns or yarn on parallel tubes, a speed of 20 to 30 r.p.m. of reel shall be used. On reels that have only one pigtail guide, the

TABLE I.

Equivalent Singles Size	Yards for Test	Conversion Formula	Number of Tests Per Case of Yarn
20's and above	60	$\frac{\text{Size}}{2}$ = Ply size	3 from each of 4 spools or tubes
3's to 20's	24	$\frac{\text{Size}}{5}$ = Ply size	3 from each of 4 spools or tubes
Below 3's	12	$\frac{\text{Size}}{10}$ = Ply size	3 from each of 4 spools or tubes

tension shall be applied by making one full wrap of the yarn around the guide. On reels using two or more guides, the yarn shall pass straight through the guides onto the reel, the angles of the guides supplying the necessary tension.

Size of All Yarns, Alternate Method.

16. All yarns used in the alternative method of testing for strength (Section 10) shall be sized under natural humidity conditions at the time of test. Plied yarns shall be prepared in skeins in accordance with Table I. The moisture regain shall then be determined as specified in Section 11, and results corrected to a common basis of standard moisture regain equal to 7 per cent of the bone-dry weight by means of the formula:

$$\text{Size corrected to standard moisture regain} = \frac{\text{Size} \times (100 + \text{actual percentage regain})}{107}$$

The average of these tests shall be the average size of case, bale, ball chain or beam warp of yarn.

TWIST

Twist of Single Yarns.

17. No precision method for determining the twist of single yarns has been developed.

Twist of Plied Yarns.

18. The ply twist in yarns of 2 or more ply shall be determined on any standard twist counter with jaws set 10 in. apart. The strands shall be clamped in jaws under a definite tension by attaching weights.

The tension to be used shall be determined from the formula:

$$\text{Tension, in grams} = \frac{156 \text{ (Constant)}}{\text{Equivalent singles size}}$$

Example.—The weight for 23/11 would be as follows:

$$\frac{156}{2.09} = 76 \text{ g.}$$

NOTE.—The constant of 156 represents a tension (basis yarn No. 1) which should be placed on yarn or cord to hold it sufficiently taut and still not remove any stretch.

Number of Tests.

19. Three twist tests on each of 4 packages of yarn from each case shall be made, and the average of these 12 tests shall be the average of the case.

STANDARD SPECIFICATIONS
FOR
TOLERANCES AND TEST METHODS FOR ELECTRICAL COTTON YARNS[1]

Serial Designation: D 203 - 25

These specifications are issued under the fixed designation D 203; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1924; ADOPTED IN AMENDED FORM, 1925.

Scope.

1. These specifications cover the tolerances and methods of testing for electrical cotton yarns, both single and multiple wound.

TOLERANCES

STRENGTH

Strength of Yarn.

2. The average tensile strength of each case or bale of yarn, either single of plied, as found by test shall be not less than the specified strength.

SIZE OR YARN NUMBER

Size of Yarn, Carded and Combed.

3. The average size of each case or bale of yarn, in the singles, either carded or combed, as found by test shall not vary more than 3 per cent above or below the specified size.

Example.—No. 36 carded yarn specified; tolerance would allow 34.92 to 37.08.

TWIST

Definition.

4. *Direction of Twist.*—The direction of twist shall be as defined in the Standard Definitions of Terms Relating to Textile Materials (Serial Designation: D 123) of the American Society for Testing Materials, as follows:

Twist, Direction of.—In the case of yarn or cord, the yarn or cord has right-hand or regular twist if when it is held vertically the spirals or twists are seen to incline upward in a right-hand direction and has left-hand or reverse twist when the spirals or twists are seen to incline upward in a left-hand direction.

NOTE.—Attention is called to the fact that this definition of twist is opposite to that used in sewing thread manufacture.

Twist of Plied Yarns.

5. The average twist of each case or bale of plied yarn as found by test shall not vary more than 5 per cent above or below that specified.

Example.—Twist specified is 20 twists per inch; tolerance 19 to 21.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

METHODS OF TESTING

STRENGTH

Testing Machine.

6. All skeins shall be broken on an automatic power yarn tester of inclination balance type of 150 and 300-lb. capacities. The lower capacity should be used until the swing of the pendulum exceeds an angle of 45 deg. from the vertical. When yarns break above this mark, the higher capacity of testing machine should be used.

Spools.

7. The spools used in holding skeins during testing shall not be less than 1 in. in diameter, and not less than 1 in. in width. The upper or head end spool should be fastened solidly, and the lower spool be allowed to revolve.

Speed of Separation.

8. The speed of pulling jaw shall be 12 in. per minute.

Reels.

9. Any yarn reel having a $1\frac{1}{2}$-yd. perimeter may be used in preparing the skeins. For yarn wound on cones, where the yarn is drawn from the top, a speed of 100 to 300 r.p.m. of reel shall be used. For yarn wound on parallel tubes, the yarn shall be drawn from the side, and a speed of 20 to 30 r.p.m. of reel shall be used. On reels that have only one pigtail guide and for yarns on cops or cones, either single end or multiple wound, the tension shall be applied by making one full wrap around the guide. For yarns on parallel tubes, either single end or multiple wound, there shall be no wraps around the guide. On reels using two or more guides, the yarn, either single end or multiple wound, shall pass straight through the guides onto the reel, the angles of the guides supplying the necessary tension. Judgment must be used in regard to the amount of tension required on yarns having little or a large amount of twist.

TABLE I.

NUMBER OF ENDS IN SINGLE STRAND	LENGTH TO REEL FOR TEST, YD.
2	60
3	45
4 and 5	30
6 and 7	21
8 and above	15

Number of Tests.

10. Three tests from each of four cops, cones or tubes of yarn from every case shall be made, and the average of these twelve tests shall be the average of the case.

Two skein test methods are given, *A*, Preferred Method and *B*, Alternate Method. The Alternate Method can be used where routine testing is done on a large scale. The Preferred Method should always be used in case of dispute.

A. Preferred Method

Strength, Single End Yarn.

11. For single end yarn a standard skein (120 yd.) shall be broken after conditioning in an atmosphere of 65 per cent relative humidity, and 70° F. (21° C.).

Strength, Multiple Wound Yarns.

12. For multiple wound yarn, a skein shall be prepared in accordance with Table I, and shall be broken after conditioning in an atmosphere of 65 per cent relative humidity, and 70° F. (21° C.). The table provides for skeins of approximately 120 yd. of single yarn or 80 loops. No skeins shall be less than 10 loops or 15 yd. of multiple strand.

Conditioning.

13. Cops, cones and tubes selected for test shall be conditioned for at least 12 hours. Yarn removed for testing after conditioning shall not penetrate into a tube a distance of more than $\frac{1}{4}$ in. on a radial line. Skeins shall be conditioned for at least 3 hours.

B. Alternate Method

Strength, Single.

14. Skeins of either single end or multiple wound yarn prepared as outlined in Method *A* shall be broken under natural humidity conditions at the time of test. The results thus obtained shall be reduced to a common basis of standard moisture regain equal to 7 per cent of the bone-dry weight.

Moisture Regain Determination.

15. To determine moisture regain present in samples, the several skeins shall be weighed collectively, immediately after testing, under natural moisture conditions which obtain at the time of test. The skeins shall then be placed in the basket of an oven at a temperature of 105 to 110° C. (221 to 230° F.) and dried to constant weight. The moisture regain is then computed as the percentage of the dry weight.

Correction to Standard Regain.

16. The following formula shall then be applied, based on the assumption that the standard moisture regain of cotton yarns is 7 per cent of the dry weight, that the actual percentage regain is between the limits of 3 and 7 per cent of the dry weight, and that for 1 per cent of moisture regain there is an increase of 6 per cent in the tensile strength of the yarn:

$$\text{Tensile strength corrected to standard moisture regain} = \frac{(\text{Tensile strength from machine reading}) \times 142}{100 + (6 \times \text{actual percentage regain})}$$

Example.—With a skein of yarn broken under natural conditions showing a tensile strength of 120 lb. as read from the machine dial and which by weighing before and after drying the skein was found to contain a moisture regain equal to 5 per cent of the bone-dry weight, the tensile strength corrected to a common basis of 7 per cent moisture regain would be:

$$\text{Corrected tensile strength} = \frac{120 \times 142}{100 + (6 \times 5)} = 131 \text{ lb.}$$

(*b*) Moisture regain tests shall be made periodically during the hours of testing as the natural humidity conditions are found to vary.

C. *Strength Correction to Size*

Strength Correction to Size.

17. The average tensile strength shall be corrected to the specified size as determined in accordance with Sections 18 and 19 by the following formula:

$$\text{Corrected tensile strength} = \text{Actual average strength} \times \frac{\text{Actual average size}}{\text{Specified size}}$$

SIZE OR YARN NUMBER

Yarn Number, Preferred Method.

18. All skeins used in the Preferred Method of testing for strength shall be sized immediately after being broken.

Yarn Number, Alternate Method.

19. All skeins used in the Alternate Method of testing for strength shall be sized under natural humidity conditions at the time of test. The moisture regain shall then be determined as outlined in Section 15 and the results corrected to a common basis of standard moisture regain equal to 7 per cent of the bone-dry weight by means of the following formula:

$$\text{Size corrected to standard moisture regain} = \frac{\text{Size} \times (100 + \text{actual percentage of regain})}{107}$$

Balance.

20. The balance to be used shall be accurate to 0.25 per cent of the standard size of the yarn. When the balance does not indicate the size directly, the yarn number or size may be calculated from the formula:

$$\text{Yarn number or size} = \frac{\text{Length in yards of single yarn}}{\text{Weight in grains}} \times \frac{7000 \text{ (grains in 1 lb.)}}{840 \text{ (yards of No. 1 cotton yarn per pound)}}$$

Number of Tests.

21. The average size of the 12 skeins used in the strength test shall be the average size of case or bale.

TWIST

Twist of Plied Yarns.

22. The ply twist in yarns of 2 or more ply shall be determined on any standard twist counter with jaws set 10 in. apart. The strands shall be clamped in jaws under a definite tension by attaching weights. The tension to be used shall be determined from the formula:

$$\text{Tension, in grams} = \frac{156 \text{ (Constant)}}{\text{Equivalent singles size}}$$

Example.—The weight for 80/2 ply would be as follows:

$$\frac{156}{40} = 3.9 \text{ g. (approximately)}$$

NOTE.—The constant of 156 represents a tension (basis yarn No. 1) which should be placed on yarn or cord to hold it sufficiently taut and still not remove any stretch.

Number of Tests.

23. Three twist tests on each of 4 packages of yarn from each case shall be made, and the average of these 12 tests shall be the average of the case.

STANDARD SPECIFICATIONS
FOR
TOLERANCES AND TEST METHODS FOR COTTON SEWING THREADS[1]

Serial Designation: D 204 - 27

These specifications are issued under the fixed designation D 204; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1924; ADOPTED IN AMENDED FORM, 1927.

TOLERANCES

STRENGTH

1. The average tensile strength of each case of sewing thread as found by test shall be not less than the specified strength. **Strength.**

YARDAGE

2. When thread is sold by weight instead of on a guaranteed yardage unit, the yards per pound shall be indicated on each tube, spool or cone. **Yards per Pound.**

3. The actual number of yards per pound or unit of sale, as found by test, shall not be more than 4 per cent less than the number of yards per pound or unit of sale specified. **Variation.**

TWIST

4. *Direction of Twist.*—In the case of sewing thread, the thread has right-hand or regular twist if when it is held vertically the spirals or twists are seen to incline upward in a left-hand direction, and has left-hand or reverse twist when the spirals or twists are seen to incline upward in a right-hand direction. **Definition.**

NOTE.—Attention is called to the fact that the definition as here given does not agree with the definition of direction of twist of yarn or cord appearing in the Standard Definitions of Terms Relating to Textile Materials (Serial Designation: D 123) of the American Society for Testing Materials,[2] which definitions define the direction of twist in such a way that left-hand or reverse twist in the case of yarn or cord corresponds to right-hand or regular twist in the case of sewing thread, as defined herein.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

[2] See p. 905.

Balance.

5. The finished sewing thread shall be balanced in twist as nearly as possible and in no case shall a yard of thread when drawn from the side of a spool or package twist upon itself more than six times when brought together to form a loop.

RUNNING QUALITIES

Running Qualities.

6. In addition to meeting the foregoing requirements, sewing thread shall be subjected to a sewing machine test under actual factory conditions. In the event of any thread apparently failing to operate properly, it shall be judged by comparison on any given machine under identical conditions, with thread from a lot known to have previously operated satisfactorily.

METHODS OF TESTING

STRENGTH

Strength.

7. All threads shall be tested by the single strand test method. A single strand tester of proper capacity with jaws set 10 in. between grips and having a speed of pulling jaw of 12 in. per minute shall be used. The average of ten breaks from each of five spools or packages shall be the average strength. Any test during which the thread breaks at the jaw due to pinching or cutting shall not be included in the average.

YARDAGE

Yards per Pound.

8. In determining the yards per pound of sewing thread, the skeins shall be prepared in accordance with Table I. Any yarn reel having a $1\frac{1}{2}$-yd. perimeter may be used in preparing the skeins. On reels that have only one pigtail guide, tension shall be applied by making one full wrap of the thread around the guide. On reels using two or more guides, the thread shall pass straight through the guides onto the reel, the angles of the guides supplying the necessary tension.

TABLE I.

EQUIVALENT SINGLE SIZE	YARDS FOR TEST	NUMBER OF TESTS PER CASE OF THREAD
Finer than 10's.....	120	3 from each of 4 spools
10's and coarser.....	60	3 from each of 4 spools

The skeins shall then be weighed and the average weight of the 12 skeins shall be converted to yards per pound from the formula:

$$\text{Yards per lb.} = \frac{7000 \times (\text{number of yards in skein})}{\text{Average weight in grains of skein}}$$

LENGTH OF PACKAGE

Length of Package.

9. (*a*) (*Preferred Method*).—The length of package of sewing thread shall be determined by multiplying the number of yards per

pound, obtained as outlined in Section 8, by the net weight in pounds of the thread in package to be tested.

(*b*) (*Alternate Method*).—The length of package of sewing thread shall be determined by measuring the entire length over a reel having a 1½-yd. perimeter, taking three full turns around the reel and delivering thread to a take-up drum. The yardage shall be noted on reel dial.

(*c*) (*Alternate Method*).—The length of package of sewing thread shall be determined by measuring the thread in the form of skeins prepared on a reel having a 1½-yd. perimeter. In no case shall the yardage of these skeins exceed that specified in Table II. The total yardage of skeins thus prepared from a package constitutes its length.

TABLE II.

EQUIVALENT SINGLE SIZE	LENGTH OF SKEIN, YD.
Finer than 12's	240
6's to 12's, inclusive	120
Coarser than 6's	60

TWIST AND BALANCE

10. No precision method for determining the twist of single yarns has been developed. **Twist in Singles.**

11. Ply twist shall be determined on any standard twist counter with the jaws set 10 in. apart. The strands shall be clamped in jaws under a definite tension obtained by attaching weights. The tension to be used shall be determined from the formula: **Twist in Ply.**

$$\text{Tension, in grams} = \frac{156 \text{ (Constant)}}{\text{Equivalent singles size}}$$

Example.—The weight for 24/4 would be as follows:

$$\frac{156}{6} = 26 \text{ g.}$$

NOTE.—The constant of 156 represents a tension (basis yarn No. 1) which should be placed on yarn or thread to hold it sufficiently taut and still not remove any stretch.

12. The "balance" of a sewing thread shall be determined by removing one yard of thread from a spool or package, holding it at each end and bringing these ends together, allowing the thread a chance to loop. **Balance.**

STANDARD SPECIFICATIONS
FOR
TOLERANCES FOR NUMBERED COTTON DUCK[1]

Serial Designation: D 230 – 27

These specifications are issued under the fixed designation D 230; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1925; ADOPTED, 1927.

Tolerances. 1. Tolerances shall be the limit within which a textile must come in its specified characteristics in order that it shall constitute a good delivery on contract. They may be classified as the allowable limits of the quantitative characteristics of the fabric as defined in the specifications. The following tolerances are based upon the Standard General Methods of Testing Woven Textile Fabrics (Serial Designation: D 39) of the American Society for Testing Materials.[2]

WIDTH

Width. 2. The average width determined by measurement shall be as specified with the following tolerances:

WIDTH, IN.	TOLERANCE, IN. UNDER	TOLERANCE, IN. OVER
Up to and including 36	1/4	1/4
37 to 60, inclusive	1/4	3/8
61 to 80, inclusive	3/8	5/8
81 to 120, inclusive	3/8	3/4

WEIGHT

Weight. 3. The weight of the fabric determined by test shall be not more than 2.5 per cent over or under the specified weight.

THREADS PER INCH

Threads per Inch. 4. (*a*) The average count of warp ends per inch determined by test shall be not more than 1½ ends over or under the specified count for fabrics counting not over 40 ends per inch and not more than 2 ends over or under for fabrics counting over 40 ends per inch.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

[2] See p. 900.

(*b*) The average count of filling picks per inch determined by test shall be not more than 1 pick over or under the specified count for fabrics counting not over 25 picks per inch, and not more than $1\frac{1}{2}$ picks over or under for fabrics counting from $25\frac{1}{2}$ to 32 picks per inch, and not more than 2 picks over or under for fabrics counting over 32 picks per inch.

TENSILE STRENGTH

5. The average tensile strength of the warp and the average tensile strength of the filling shall be not less than the average specified tensile strength of each. Tensile Strength.

STANDARD SPECIFICATIONS

FOR

TOLERANCES AND TEST METHODS FOR TIRE FABRICS OTHER THAN CORD FABRICS[1]

Serial Designation: D 122 – 27

These specifications are issued under the fixed designation D 122; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1925; REVISED, 1927.

TOLERANCES

Tolerances.

1. Tolerances shall be the limits within which a textile must come in its specified characteristics in order that it shall constitute a good delivery on contract. They may be classified as the allowable limits of the quantitative characteristics of the fabric as defined in the specifications.

WIDTH

Width.

2. The average width determined by measurements shall not be more than 1 per cent over or under the specified width.

WEIGHT

Weight per Square Yard.

3. The weight per square yard of the roll or piece determined by test shall not be more than:

(*a*) Two and one-half per cent over or under the specified weight per square yard for fabrics over 11 oz. per sq. yd. in weight.

(*b*) Three per cent over or under the specified weight per square yard for fabrics 11 oz. per sq. yd. or lighter.

COUNT

Count per Inch.

4. (*a*) The average count of the warp ends per inch determined by test shall not be more than 1 per cent over or under the specified count.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

(*b*) The average count of the filling picks per inch determined by test shall not vary from the specified count more than:

± ½ pick for fabrics counting	not over 25 picks per inch
± 1 pick for fabrics counting	25½ to 32 picks per inch
± 1½ picks for fabrics counting	over 32 picks per inch

THICKNESS (GAGE)

Thickness (Gage).

5. The average thickness of the fabric determined by test shall not be more than 4 per cent over or under the specified thickness in fabrics made of the combed staple, and not more than 5 per cent over or under the specified thickness in fabrics made of the carded staple.

TENSILE STRENGTH

Tensile Strength.

6. The average tensile strength of the warp and the average tensile strength of the filling determined by test shall not be less than the specified tensile strength.

TWIST

Twist.

7. The average ply twist determined by test shall not be more than 5 per cent over or under the specified twist.

CRIMP

Crimp.

8. (*a*) The average warp crimp shall not be more than 10 per cent over or under the specified percentage of crimp.

(*b*) The average filling crimp shall not be more than 20 per cent over or under the specified percentage of crimp.

BOW

Bow.

9. The average bow determined by test shall not be more than 1 per cent of the width of the fabric in inches over or under the specified bow.

METHODS OF TESTING[1]

WIDTH

Width.

10. The average width shall be determined by measuring at five different places uniformly distributed along the full length of the roll or piece. No measurement shall be considered within 3 yards of either end of the roll or piece.

WEIGHT

Weight per Square Yard.

11. The net weight of the roll or piece from which the covering or binding has been removed shall be determined.

[1] All test procedure not covered in these specifications shall be in accordance with the Standard General Methods of Testing Woven Textile Fabrics (Serial Designation: D 39) of the American Society for Testing Materials, see p. 900.

The net weight of the roll or piece divided by its standard area in square yards shall be taken as the normal net weight per square yard.

COUNT

Count. 12. The average number of ends and picks per inch of the fabric shall be determined by counting a space not less than 1 in. in five different places uniformly distributed along the full length of the roll or piece. No count shall be considered within 3 yards of either end of the roll or piece.

THICKNESS (GAGE)

Thickness (Gage). 13. The average thickness of the fabric shall be determined by means of an automatic micrometer equipped with a foot $\frac{3}{8}$ in. in diameter. The foot of the micrometer shall be raised $\frac{1}{4}$ in. above the fabric and allowed to drop under a dead weight pressure of 6 oz. The gage shall be taken in five different places uniformly distributed along the full length of the roll or piece. No gage shall be considered within 6 in. of the selvages nor within 3 yards of either end of the roll or piece.

TENSILE STRENGTH

Tensile Strength. 14. The average tensile strength shall be determined by testing five samples each of warp and filling on any standard type of inclination balance machine of 400 and 800-lb. capacities.

The lower capacity of the testing machine should be used until the swing of the pendulum exceeds an angle of 45 deg. from the vertical. When the fabric breaks above this mark, the higher capacity of the testing machine shall be used. The speed of the pulling jaw shall be 20 in. per minute. The length of the test specimen between nip of jaws shall be 3 in.

TWIST

Twist. 15. The ply twist of warp or filling shall be based on ten tests, each taken on any standard twist counter with the jaws set 10 in. apart. Each test specimen shall be clamped in the jaws under a definite tension by attaching weights. The tension to be used shall be determined from the formula:

$$\text{Tension (in grams)} = \frac{156 \text{ (Constant)}}{\text{Theoretical yarn size}}$$

Example.—The weight to be attached for 23/11 ply would be as follows:

$$\frac{156}{2.09} = 76 \text{ g. (approximate)}$$

NOTE.—The constant of 156 represents a tension (basis size No. 1 yarn) which should be placed on the cord to hold it sufficiently taut and still not remove any stretch.

CRIMP

16. The average crimp of warp or filling shall be determined by testing at least four adjacent samples each of warp and filling yarns under standard conditions. Crimp.

The yarns for test shall be taken from the inside edge of the test piece as near the center line of the roll as possible.

The yarns shall then be straightened by a machine which applies a definite tension by attaching weights. The tension to be used shall be determined from the formula:

$$\text{Tension (in grams)} = \frac{156 \text{ (Constant)}}{\text{Theoretical yarn size}}$$

The machine shall be equipped with a dial or scale so arranged that the length between the original markings shall be read in terms of percentage elongation or crimp.

BOW

17. The average bow shall be determined by testing the roll or piece in at least three places. The tests shall be made preferably at points 10 yards from each end of the roll or piece and in the center. Bow.

STANDARD SPECIFICATIONS
FOR
TOLERANCES AND TEST METHODS FOR CORD TIRE FABRICS[1]

Serial Designation: D 179 – 27

These specifications are issued under the fixed designation D 179; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED IN AMENDED FORM, 1925; REVISED, 1927.

TOLERANCES

Tolerances.

1. Tolerances shall be the limits within which a textile must come in its specified characteristics in order that it shall constitute a good delivery on contract. They may be classified as the allowable limits of the quantitative characteristics of the fabric as defined in the specifications.

WIDTH

Width.

2. The average width determined by measurements shall not be more than 1.25 per cent over or under the specified width.

WEIGHT

Weight per Square Yard.

3. The weight per square yard of the roll or piece determined by test shall not be more than 3 per cent over or under the specified weight per square yard.

COUNT

Count per Inch.

4. (*a*) The average count of the warp ends per inch determined by test shall not be more than 1.25 per cent over or under the specified count.

(*b*) The average count per inch of the filling picks determined by test shall not be more than 10 per cent over or under the specified count.

THICKNESS (GAGE)

Thickness (Gage).

5. The average thickness of the warp cords determined by test shall not be more than 3 per cent under or over the specified thickness

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

in cords made of combed staple, and not more than 3.5 per cent under or over the specified thickness in cords made of carded staple.

TENSILE STRENGTH

6. The average tensile strength of the cord found by test shall not be less than the specified tensile strength. **Tensile Strength.**

ELONGATION

7. The average percentage of elongation of the cord determined by test shall not be more than 10 per cent over or under the specified elongation at either a definite load or the ultimate breaking point. **Elongation.**

TWIST

8. (*a*) The average twist of the plied yarn determined by test shall not be more than 5 per cent over or under the specified twist. **Twist.**

(*b*) The average twist of the cabled cord determined by test shall not be more than 4 per cent over or under the specified twist.

METHODS OF TESTING[1]

WIDTH

9. The average width shall be determined by measuring at five different places uniformly distributed along the full length of the roll or piece. Each determination shall be made with the material laid flat on the measuring table and the tension released. No measurement shall be considered within 3 yards of either end of the roll or piece. **Width.**

WEIGHT

10. The net weight of the roll or piece from which the covering or binding has been removed shall be determined. **Weight per Square Yard.**

The net weight of the roll or piece divided by its standard area in square yards shall be taken as the normal net weight per square yard.

COUNT

11. The average number of warp ends per inch of the fabric shall be determined by counting a space of not less than 1 in. in five places across the width, that is, at the center and at each side, but not nearer than $\frac{1}{4}$ in. from the selvage, and repeating this in five different places uniformly distributed along the full length of the roll or piece. No count shall be considered within 3 yards of either end of the roll or piece. **Count Warp.**

12. The average number of picks per inch of the fabric shall be determined by counting a space of not less than 2 in. in five different **Count Filling.**

[1] All test procedure not covered in these specifications shall be in accordance with the Standard General Methods of Testing Woven Textile Fabrics (Serial Designation: D 39) of the American Society for Testing Materials, see p. 900.

places uniformly distributed along the full length of the roll or piece. No count shall be considered within 3 yards of either end of the roll or piece.

THICKNESS (GAGE)

Thickness (Gage).

13. The thickness of the warp cords shall be determined by an automatic micrometer equipped with a foot $\frac{3}{8}$ in. in diameter. The filling picks shall be displaced so as to allow four warp cords to be placed side by side under the foot of the micrometer which shall then be raised $\frac{1}{4}$ in. and allowed to drop with a dead weight pressure of 6 oz. The gage shall be taken in five different places uniformly distributed along the full length of the roll or piece. No gage shall be considered within 3 yards of either end of the roll or piece.

TENSILE STRENGTH

Tensile Strength.

14. The average tensile strength shall be determined by testing at least ten cords on a single cord tester of 25 and 50-lb. capacity. The lower capacity of the testing machine should be used until the swing of the pendulum exceeds an angle of 45 deg. from the vertical. When cords break above this mark, the higher capacity of the testing machine shall be used. The speed of the pulling jaw shall be 12 in. per minute. The length of the test specimen between nip of jaws, measured along the line of the cord, shall be 10 in.

ELONGATION

Elongation.

15. The average elongation shall be determined in conjunction with the tensile strength tests and preferably by means of an autographic recorder. Each cord at the time of test shall be subjected to an initial tension of 4 oz., dead weight, before locking it in the bottom jaw.

TWIST

Twist.

16. The average twist of both ply and cable shall be based on ten tests each taken on any standard twist counter with the jaws set 10 in. apart. Each test specimen shall be clamped in the jaws under a definite tension by attaching weights. The tension to be used shall be determined from the formula:

$$\text{Tension (in grams)} = \frac{156 \text{ (Constant)}}{\text{Theoretical yarn size}}$$

Example.—The weight to be attached for 23/5/3 ply would be as follows:

$$\frac{156}{1.53} = 102 \text{ g. (approximate)}$$

NOTE.—The constant of 156 represents a tension (basis size No. 1 yarn) which should be placed on the cord to hold it sufficiently taut and still not remove any stretch.

STANDARD SPECIFICATIONS

FOR

TOLERANCES FOR HOSE DUCKS AND BELT DUCKS[1]

Serial Designation: D 181 - 25

These specifications are issued under the fixed designation D 181; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED IN AMENDED FORM, 1925.

Tolerances.

1. Tolerances shall be the limit within which a textile must come in its specified characteristics in order that it shall constitute a good delivery on contract. They may be classified as the allowable limits of the quantitative characteristics of the fabric as defined in the specifications. The following tolerances are based upon the Standard General Methods of Testing Woven Textile Fabrics (Serial Designation: D 39) of the American Society for Testing Materials.[2]

WIDTH

Hose Ducks.

2. *Hose Ducks*, ranging in width from 40 to 72 in. inclusive:

(*a*) No individual measurement made to determine the width of the fabric shall be more than 2 per cent over or under the specified average width.

(*b*) The average width determined by measurements shall be not more than 1½ per cent over or under the specified average width.

Rubber and Balata Ducks.

3. *Rubber and Balata Belt Ducks*, ranging in width from 42 to 72 in., inclusive:

(*a*) No individual measurement made to determine the width of the fabric shall be more than 1½ per cent over or under the specified average width.

(*b*) The average width determined by measurements shall be not more than 1 per cent over or under the specified average width.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

[2] See p. 900.

Stitched Canvas Belting Ducks.

4. *Stitched Canvas Belting Ducks*, ranging in width from 16 to 72 in., inclusive:

(*a*) No individual measurement made to determine the width of the fabric shall be more than $\frac{1}{8}$ in. over or under the tolerance for the average width.

(*b*) The average width determined by measurement shall be as specified with the following tolerances:

Width, in.	Tolerance, in. Under	Over
16 to 36, inclusive	$\frac{1}{4}$	$\frac{1}{4}$
37 to 60, inclusive	$\frac{1}{4}$	$\frac{3}{8}$
61 to 72, inclusive	$\frac{3}{8}$	$\frac{5}{8}$

WEIGHT

Normal Weight.

5. The weight of the fabric determined by test shall be not more than $2\frac{1}{2}$ per cent over or under the specified weight.

THREADS PER INCH

Hose Ducks.

6. *Hose Ducks.*—

(*a*) The average count of warp ends per inch determined by test shall be not more than 2 per cent over or under the specified count.

(*b*) The average count of filling picks per inch determined by test shall be not more than 4 per cent over or under the specified count.

Rubber Belt Ducks.

7. *Rubber Belt Ducks.*—

(*a*) The average count of warp ends per inch determined by test shall be not more than 2 per cent over or under the specified count.

(*b*) The average count of filling picks per inch determined by test shall be not more than 4 per cent over or under the specified count.

Stitched Canvas Belting Ducks.

8. *Stitched Canvas Belting Ducks.*—

(*a*) The average count of warp ends per inch determined by test shall be not more than 1 end over or under the specified count for fabrics counting not over 30 ends per inch and not more than $1\frac{1}{2}$ ends over or under for fabrics counting over 30 ends per inch.

(*b*) The average count of filling picks per inch determined by test shall be not more than 1 pick over or under the specified count for fabrics counting not over 15 picks per inch, and not more than $1\frac{1}{2}$ picks over or under for fabrics counting over 15 picks per inch.

GAGE

Hose Ducks.

9. *Hose Ducks.*—The average thickness of the fabric shall be not more than 7 per cent over or under the specified average thickness.

10. *Rubber and Balata Belt Ducks.*—The average thickness of the fabric shall be not more than 5 per cent over or under the specified average thickness. Rubber and Balata Ducks.

CRIMP

11. The average warp crimp shall not be more than 10 per cent over or under the specified crimp percentage, and the average filling crimp shall not be more than 20 per cent over or under the specified crimp percentage. Crimp.

TENSILE STRENGTH

12. The average tensile strength of the warp and the average tensile strength of the filling shall be not less than the average specified tensile strength. Tensile Strength.

STANDARD SPECIFICATIONS
FOR
TOLERANCES AND TEST METHODS FOR ELECTRICAL SILK AND COTTON TAPES[1]

Serial Designation: D 259 – 27

These specifications are issued under the fixed designation D 259; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1926; ADOPTED, 1927.

Scope.

1. These specifications cover the tolerances and test methods for silk and cotton tape used in the electrical industry for purposes of insulation.

TOLERANCES

THICKNESS

Thickness.

2. The thickness of the fabric shall not vary from that specified by more than the following amounts:

THICKNESS, IN.	PERMISSIBLE VARIATIONS
Up to and including 0.007	±0.0008 in.
Over 0.007 and including 0.011	±0.001 in.
Over 0.011 and including 0.020	±0.0015 in.

WIDTH

Width.

3. The width of the fabric shall not vary from that specified by more than the following amounts:

NOMINAL WIDTH OF FABRIC, IN.	PERMISSIBLE VARIATIONS
$\frac{1}{2}$	$\pm\frac{1}{32}$ in.
$\frac{3}{4}$	$\pm\frac{1}{32}$ in.
1	$\pm\frac{3}{64}$ in.
$1\frac{1}{4}$	$\pm\frac{3}{64}$ in.
$1\frac{1}{2}$	$\pm\frac{3}{64}$ in.
Above $1\frac{1}{2}$	$\pm\frac{1}{16}$ in.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

WEIGHT

4. The average weight of the fabric shall be calculated in pounds per gross yards and in no case shall the weight exceed the agreed specified weight by more than 3 per cent. **Weight.**

CONSTRUCTION

5. The total number of ends shall be not less than specified. The picks per inch shall not vary more than: **Construction.**

±1 pick for tapes counting up to and including 48 per inch;

±2 picks for tapes counting over 48 per inch.

METHODS OF TESTING

THICKNESS

6. The thickness of the fabric may be determined by the use of any accurate dial gage measuring device acceptable to both supplier and user or by the use of micrometers having measuring faces not less than $\frac{7}{16}$ in. in diameter or more than $\frac{1}{2}$ in. in diameter. **Method of Measuring Thickness.**

7. The range of thickness shall be recorded on the sample used for the weight determination hereinafter described when using an indicating device. When ordinary micrometers are used, at least twenty measurements should be taken on the same sample and the range of thickness recorded. **Number of Measurements.**

WIDTH

8. The width of the narrow fabric shall be determined by the use of a standard steel scale graduated to read within the degree of accuracy referred to in the table of tolerances. The fabric shall lay flat on a smooth surface at the time of measurement. **Method of Measuring Width.**

9. The same number of measurements are to be made on the same length specimens as in the case of the thickness. **Number of Measurements.**

WEIGHT

10. The weight of the fabric shall be determined by accurately measuring a sample, not less than 12 yd. in length, either by hand or by the use of any accurate measuring device and then weighing the sample and calculating the weight in pounds per gross yards. **Weight.**

11. One weight determination shall be made per gross yards. The average weight of all tape tested shall indicate the average weight of the shipment involved. **Number of Tests.**

CONSTRUCTION

12. The total number of ends shall be determined irrespective of the width of the fabric. **Ends.**

Picks.

13. The number of picks shall be determined in a length of 1 in.

Number of Tests.

14. Both the number of ends and number of picks shall be taken as the average of five counts on five different places on the sample. An accepted thread counting magnifying glass shall be used in making these counts.

TENSILE STRENGTH

Tensile Strength.

15. The tensile strength of the tape shall be determined on a testing machine which complies with the Standard Specifications for Textile Testing Machines (Serial Designation: D 76) of the American Society for Testing Materials,[1] and tests shall be made in the direction of warp only.

Number of Tests.

16. The specimens shall be tested in full section and the distance between jaws shall be 3 in.

NOTE.—In case of dispute, tests shall be made on material under standard conditions as defined in Section 3 of the Standard General Methods of Testing Woven Textile Fabrics (Serial Designation: D 39) of the American Society for Testing Materials.[2]

Number of Rolls Tested.

17. At least five strength determinations shall be made and the average of these five tests are to indicate the strength of the roll.

NUMBER OF TESTS

Number of Tests.

18. From each shipment of electrical cotton tape at least three rolls shall be selected and the tests outlined shall be made on each roll. Failure of the first samples selected to conform to the requirements of the specifications will necessitate a selection of the same number of rolls for a second test.

Failure.

19. Failure of any of the tape to conform to the requirements of these specifications will make the shipment the subject of complaint and disposition in accordance with the agreement between the supplier and user.

[1] See p. 867.

[2] See p. 900.

STANDARD SPECIFICATIONS AND TESTS

FOR

OSNABURG CEMENT SACKS[1]

Serial Designation: D 205 – 27

These specifications are issued under the fixed designation D 205; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1924; ADOPTED IN AMENDED FORM, 1925; REVISED, 1927.

Standard Methods.

1. All test procedure not covered in these specifications shall be in accordance with the Standard General Methods of Testing Woven Textile Fabrics (Serial Designation: D 39) of the American Society for Testing Materials.[2]

Tolerances.

2. Tolerances shall be the limits within which the fabric or sacks must come in their specified characteristics in order that they shall constitute a good delivery on contract.

SAMPLING

Sampling.

3. Ten bales shall be taken at random from each carload of sacks and one sack shall be taken for test purposes from each of these bales. Test sacks shall be selected and tested promptly upon receipt of shipment.

REQUIREMENTS AND METHODS OF TEST

A. Material (Osnaburg)

Width.

4. The width when measured at right angles to the selvage with the material smoothed out flat with only sufficient tension to remove wrinkles shall not be less than 29½ in. nor more than 31 in., the desired width being 30 in.

Weight.

5. (*a*) The weight of the material shall be 7 oz. per lineal yard (8.4 oz. per sq. yd.). A tolerance of 2 per cent under the 7-oz. requirement will be permitted.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

[2] See p. 900.

(*b*) The weight shall be determined upon material having normal moisture content obtained by exposure for at least 4 hours to an atmospheric condition of 65-per-cent relative humidity at a temperature of 21° C. (70° F.). (Note 1.)

Thread Count. 6. The thread count shall be 38 or more per inch in the warp and 28 or more per inch in the filling. A tolerance of one thread under in either warp or filling will be permitted.

Tensile Strength. 7. (*a*) The tensile strength of the warp and filling shall each average not more than 5 lb. under that specified by the purchaser.

(*b*) The tensile strength shall be determined on an approved type of inclination balance machine, motor driven. The test shall be made upon material having a normal moisture content obtained by exposure for at least 4 hours to an atmospheric condition of 65-per-cent relative humidity at a temperature of 21° C. (70° F.). (Note 1.)

Sizing. 8. No sizing or loading compound that is known to be injurious to the material or that will materially affect its weight or strength shall be used.

B. Sacks

Cut. 9. (*a*) The cut of the sacks shall average that specified by the purchaser and shall be measured as follows: Sacks with seams ripped out shall be subjected to standard atmosphere for 2 hours under a tension of 16 oz. per in. of width. Measurement shall be made at the end of this period while the sacks are still under this atmospheric condition and tension. (Note 2.)

(*b*) If the cut averages less than, but within $\frac{1}{2}$ in. of that specified, the shortage shall be subject to adjustment between the seller and purchaser.

(*c*) If the cut averages more than $\frac{1}{2}$ in. less than that specified, the sacks may be rejected.

Seams. 10. The seams shall be $\frac{1}{2}$ in. sewed with a two-thread stitch with not less than 7 stitches to 2 in. The thread shall be No. 10 yarn of three and five-ply or its equivalent in strength of other ply and number.

Hem, Printing, Valve Size. 11. The hem, printing, and size of the valve of the sack shall all be as specified by the purchaser.

RETESTING

Retest. 12. (*a*) Should the test sacks fail to conform to the requirements specified, a retest shall be made.

(*b*) Three sacks for retest shall be taken in the presence of the seller's representative from each of ten other bales taken at random; one sack of each set of three to be retained by the seller, one by the buyer, and one to be submitted to a nationally known laboratory satisfactory to both parties, whose results shall be the basis of settlement.

EXPLANATORY NOTES

NOTE 1.—Referring to sections 5 (*b*) and 7 (*b*), it is recommended that routine tests for weight and tensile strength be made under prevailing atmospheric conditions. Should results indicate unsatisfactory weight or tensile strength, the results of tests made in accordance with the specifications shall govern.

NOTE 2.—Referring to section 9 (*a*), it is recommended that routine tests for cut of sacks be made with seams ripped out and material folded down the center parallel to the selvage; the measurement to be made along the fold with only sufficient tension on the material to smooth out wrinkles. Should the results indicate unsatisfactory cut, the results of tests made in accordance with the specifications shall govern.

STANDARD GENERAL METHODS

OF

TESTING WOVEN TEXTILE FABRICS[1]

Serial Designation: D 39 – 27

These methods are issued under the fixed designation D 39; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1915; ADOPTED IN AMENDED FORM, 1920; REVISED, 1924, 1927.

GENERAL TEST METHODS

Scope.

1. These General Methods of Testing are intended to be applicable for testing woven textile fabrics. Where a material requires special treatment, specific methods of test will be described applicable for that material and such special methods shall take precedence over the general methods. The cord fabric used in manufacturing tires is not considered to be a "woven" fabric within the meaning of these methods.

CONDITION

Dry Condition.

2. The dry condition of woven textile fabrics shall be understood to be absolute dryness obtained by placing the material in a ventilated drying oven maintained at a temperature of from 100 to 110° C. (212 to 230° F.) and drying to constant weight as determined by two consecutive weighings without removal from the oven, to be taken not less than ten minutes apart, and showing a further loss of not more than 0.1 per cent from the previous weighing.

Standard Condition.

3. The standard condition of woven textile fabric shall be understood to be the condition which it assumes on exposure for at least four hours, to an atmosphere having a relative humidity of 65 per cent at 70° F. (21.1° C.).

Test Condition.

4. All fabrics shall be tested under standard condition as described in Section 3, unless the bone-dry condition, described in Section 2, is specified in the purchase specifications covering a particular contract.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

TEST METHODS

(*A*) *Length*

Length. 5. (*a*) If the fabric weighs more than 6 oz. per sq. yd., the length of a roll or piece shall be determined by running the cloth over a measuring drum of known circumference, from which the yardage is registered by a dial or counter driven by a chain, or other positive or non-slip mechanism. Just enough uniform tension[1] shall be used on the cloth to keep it running flat and true.

(*b*) If the fabric weighs 6 oz. per sq. yd. or less, its length shall be determined by running it through a folding device which will fold a known length of the fabric at each stroke. The yardage shall be registered by a dial or counter which shall be accurately calibrated against the length of the fabric per stroke.

(*B*) *Width*

Width. 6. (*a*) The width of a roll or piece shall be determined at five different places uniformly distributed along the full length of the roll or piece, and may be determined at the same time as the total length.

(*b*) Measurements shall be made to the nearest $\frac{1}{16}$ in.

(*c*) The average of the five measurements shall be the width.

(*C*) *Weight*

Weight. 7. A sample (or samples) for the weight test, containing in the aggregate not less than 20 sq. in., shall be stamped or cut from the fabric in such a way as to be fairly representative of the material. The sample (or samples) shall be conditioned as specified in Sections 2 or 3, and weighed on a balance sensitive to 0.1 per cent of the total weight.

Moisture Content and Regain. 8. The difference between the normal weight of the fabric and the dry weight is the amount of moisture present.

(*a*) When computed as percentage of the normal weight, this is the "Moisture Content" of the material.

(*b*) When computed as percentage of the dry weight, this is the "Moisture Regain" of the material.

(*D*) *Number of Threads per Inch*

Number of Threads per Inch. 9. (*a*) Where there are more than 25 threads per inch, the number of threads per inch or count of the fabric shall be determined by counting a space of not less than 1 in., in at least five different places

[1] It has been suggested that a uniform tension of two and one-half times the weight of five running yards of the fabric will be sufficient to keep the fabric flat and preserve a proper relation for comparison of different fabrics. Invitation is extended to report the results of such tests upon different fabrics.

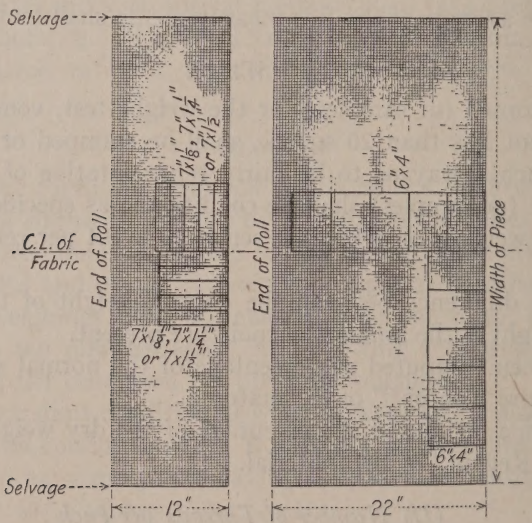
in the roll or piece. Where there are 25 or fewer threads per inch, a 3-in. gage length shall be used.

(*b*) For fabrics 3 in. in width or less, the entire width shall be used in counting the warp threads; for wider fabrics, the gage length for counting the warp threads shall be at least 1 in. from the selvage.

Count. (*c*) The average of five determinations shall be the count.

TABLE I.—TENSILE STRENGTH TEST METHODS.

Method No.	1 Strip Method	2 Grab Method
Length of specimens, in.	7	6
Width of specimens, in.:		
If thread count is 80 or over	$1\frac{1}{8}$	4
If thread count is 50 to 80	$1\frac{1}{4}$	
If thread count is less than 50	$1\frac{1}{2}$	
Ravelled to standard or specified threads per — in.	1	..
Minimum width of bottom or back jaws, in.	$1\frac{1}{2}$	2
Width of top or front jaws, in.	$1\frac{1}{2}$ (min.)	1
Distance between jaws, in.	3	3
Speed of pulling jaw, in. per minute	12	12
Number of specimens each, warp and filling	5	5



No. 1 No. 2

(*E*) *Thickness*

Thickness. 10. (*a*) The thickness shall be measured by an automatic micrometer which presses upon a circle $\frac{3}{8}$ in. in diameter of the fabric

with a pressure of 6 oz., and which is so mounted as to make possible measurements 6 in. from the selvage. All thickness measurements shall be exclusive of the selvage.

(*b*) At least five measurements at different portions of the roll or piece shall be made, and the average shall be the thickness of the fabric.

(*F*) *Tensile Strength*

Tensile Strength.

11. Tensile strength tests shall be made by one of the two methods as described in detail in Section 12. In each case the results shall be recorded separately for warp and filling.

Test Specimens.

12. For the determination of tensile strength a swatch of sufficient length as specified in Table I shall be cut the entire width of the cloth, and ten test specimens shall be cut from this swatch, five with

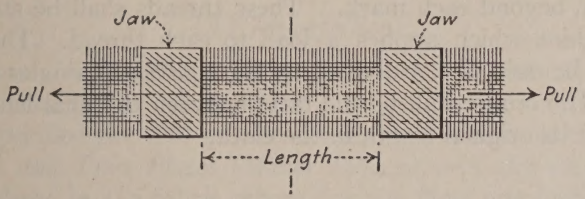


FIG. 1.—Illustration of Strip Test.

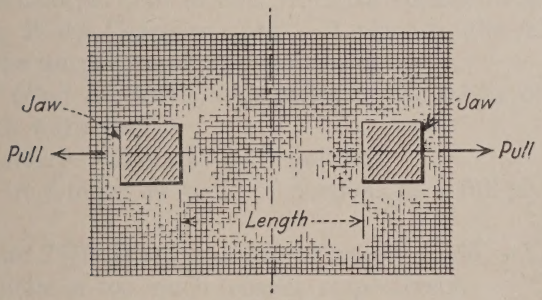


FIG. 2.—Illustration of Grab Test.

the long dimension in the direction of the warp and five in the direction of the filling. The specimens shall be fairly representative of the material. Except in narrow fabrics (less than 3 in. in width), all specimens shall be taken at least 1 in. from the selvage.

Strip Test.

(*a*) *Strip Test* (*Fig. 1*).—Under this method a long narrow strip of the fabric shall be clamped at each end by the jaws of the testing machine and strained to the point of rupture.

Grab Test.

(*b*) *Grab Test* (*Fig. 2*).—Under this method the testing machine jaws, each of definite width, shall be made to reach into the body of a rectangular piece of fabric and shall be clamped a definite distance

apart. Care shall be taken that the same set of threads are embraced by both pairs of jaws. The specimen shall then be strained to the point of rupture.

(*c*) All fabrics shall be tested by the grab method, Paragraph (*b*), unless the strip method, Paragraph (*a*) is specified in the purchase specifications covering a particular contract.

(*d*) The average of the five individual tests on the warp shall be the strength of the warp and the average of the five individual tests on the filling shall be the strength of the filling.

(*G*) *Crimp*

Crimp.

13. Two parallel lines shall be marked on the fabric at least 6 in. apart. Four threads shall be ravelled out, long enough to extend about 2 in. beyond each mark. These threads shall be straightened by a machine which applies a load to each thread. The load (in grams) to be used is 156 divided by the equivalent singles size of the thread. The crimp shall be the ratio between the final length of the thread and its original length in the cloth.

STANDARD DEFINITIONS OF TERMS RELATING TO TEXTILE MATERIALS[1]

Serial Designation: D 123 - 27

These definitions are issued under the fixed designation D 123; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1927.

(*A*) *Imperfections*

Thick and Thin Places (Major Defects).—In the case of major defects, places in the fabric where for one inch or more the count varies a specified per cent or over from the specified count.

Thick and Thin Places (Minor Defects).—In the case of minor defects, places in the fabric where for less than one inch the count varies a specified per cent or over from the specified count.

Missing Warp (Yarn or Cord).—A place in the fabric where a portion of the warp yarn or cord is missing.

Pucker (Puff or Blister).—A series in the fabric of six or more tight or slack warp yarns.

Bad Start-up.—Requires no definition.

Knot.—A joining by tying of portions of a full-ply yarn in the fabric.

Slack and Tight Warp or Filling Yarns or Cords.—Yarns or cords having too little or too much tension, respectively.

Slug.—A bunch of lint entangled in the yarn, cord or fabric.

Sewed Yarn.—A yarn in the fabric interwoven by hand.

Corkscrew Twist.—A place in the yarn or cord where an uneven twist gives a corkscrew-like appearance.

Mispick or Broken Pick.—A streak across the width of the fabric caused by a missing or partly missing pick.

Float.—A place in the fabric where warp and filling threads are incorrectly interlaced.

Kink.—A place in the fabric where a slack strand twists on itself, forming a loop on the yarn or cord.

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[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

Reed Mark.—Marks or lines up the warp way of the cloth, usually caused by too many warp yarns being drawn into a dent, a faulty setting of the loom or imperfections in the reed.

Split or Chafed Yarn or Cord.—One or more ends of ply yarn or cord broken in the fabric and chafed into a bunch.

Dirty Yarn.—Requires no definition.

Wrong Ply Warp (Yarn or Cord).—Requires no definition.

Wrong Draw.—A streak lengthwise of the fabric where the filling floats over more than one yarn due to a warp yarn having been drawn through the wrong harness.

Oil Spot.—Requires no definition.

Mixed Yarn of Inferior Grade.—Requires no definition.

Irregular Pick.—A place in the fabric where the picks are irregularly spaced.

Smash.—A place in the fabric where the warp, yarns, or cords have been broken due to the shuttle getting caught in the shed during weaving.

Imperfect Selvage.—Tight, slack, broken, weak, nicked, or open selvage.

Baggy Fabric.—A loose place in the fabric which gives the appearance of a bag or pocket.

Wrong Ply Filling Yarn.—Requires no definition.

Mixed Yarn or Cord.—Requires no definition.

Defective Splice.—A splice having irregular final twist, untrimmed ends or excessive thickness.

(*B*) *Mechanical Cotton Fabric*

The term "Mechanical Fabric" shall be understood to mean fabric manufactured for use as an intermediate product in the making of some mechanically constructed article.

NOTE.—Mechanical fabrics shall be referred to by the following descriptive nomenclature:

A word or phrase generally descriptive of the appearance of the material, or the use to which it is put.

Weight in ounces per square yard. The words "oz. sq. yd." are to be written after the weight. To avoid confusion in regard to weight of material, it is suggested that immediately succeeding the square yard weight, the weight of the material on whatever basis may have been used previous to the adoption of these standards be inserted in brackets. Table I gives the conversion values of linear yard weight to square yard weight.

Width in inches.

Count, giving first the ends, and second the picks per inch.

Example: Hose Duck, 9 oz. sq. yd. [40 in. 10 oz.], 48 in., 28 by 18.

Table I.—Conversion Table of Linear Yard Weight to Square Yard Weight of Fabric.

Weight, oz.	Width, in.																														
	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60
8.....	9.60	9.29	9.00	8.73	8.47	8.23	8.00	7.78	7.58	7.38	7.20	7.02	6.86	6.70	6.55	6.40	6.26	6.13	6.00	5.88	5.76	5.65	5.54	5.43	5.33	5.24	5.14	5.05	4.97	4.88	4.80
9.....	10.80	10.45	10.13	9.82	9.53	9.26	9.00	8.76	8.53	8.31	8.10	7.90	7.71	7.53	7.36	7.20	7.04	6.89	6.75	6.61	6.48	6.35	6.23	6.11	6.00	5.89	5.79	5.68	5.59	5.49	5.40
10.....	12.00	11.61	11.25	10.91	10.59	10.29	10.00	9.73	9.47	9.23	9.00	8.78	8.57	8.37	8.18	8.00	7.83	7.66	7.50	7.35	7.20	7.06	6.92	6.79	6.67	6.55	6.43	6.32	6.21	6.10	6.00
11.....	13.20	12.77	12.38	12.00	11.65	11.31	11.00	10.70	10.42	10.15	9.90	9.66	9.43	9.21	9.00	8.80	8.61	8.43	8.25	8.08	7.92	7.76	7.62	7.47	7.33	7.20	7.07	6.95	6.83	6.71	6.60
12.....	14.40	13.94	13.50	13.09	12.71	12.34	12.00	11.68	11.37	11.08	10.80	10.54	10.29	10.05	9.82	9.60	9.39	9.19	9.00	8.82	8.64	8.47	8.31	8.15	8.00	7.85	7.71	7.58	7.45	7.32	7.20
13.....	15.60	15.10	14.63	14.18	13.76	13.37	13.00	12.65	12.32	12.00	11.70	11.41	11.14	10.88	10.64	10.40	10.17	9.96	9.75	9.55	9.36	9.18	9.00	8.83	8.67	8.51	8.36	8.21	8.07	7.93	7.80
14.....	16.80	16.26	15.75	15.27	14.82	14.40	14.00	13.62	13.26	12.92	12.60	12.29	12.00	11.72	11.45	11.20	10.96	10.72	10.50	10.29	10.08	9.88	9.69	9.51	9.33	9.16	9.00	8.84	8.69	8.54	8.40
15.....	18.00	17.42	16.88	16.36	15.88	15.43	15.00	14.60	14.21	13.85	13.50	13.17	12.86	12.56	12.27	12.00	11.74	11.49	11.25	11.02	10.80	10.59	10.38	10.19	10.00	9.82	9.64	9.47	9.31	9.15	9.00
16.....	19.20	18.58	18.00	17.45	16.94	16.46	16.00	15.57	15.16	14.77	14.40	14.05	13.71	13.40	13.09	12.80	12.52	12.26	12.00	11.76	11.52	11.29	11.08	10.87	10.67	10.47	10.29	10.11	9.93	9.76	9.60
17.....	20.40	19.74	19.13	18.56	18.00	17.49	17.00	16.54	16.11	15.69	15.30	14.93	14.57	14.23	13.91	13.60	13.30	13.02	12.75	12.49	12.24	12.00	11.77	11.55	11.33	11.13	10.93	10.74	10.55	10.37	10.20
18.....	21.60	20.90	20.25	19.64	19.06	18.51	18.00	17.51	17.05	16.62	16.20	15.80	15.43	15.07	14.73	14.40	14.09	13.79	13.50	13.22	12.96	12.71	12.46	12.23	12.00	11.78	11.57	11.37	11.17	10.98	10.80
19.....	22.80	22.06	21.38	20.73	20.12	19.54	19.00	18.49	18.10	17.54	17.10	16.68	16.29	15.91	15.55	15.20	14.87	14.55	14.25	13.96	13.68	13.41	13.15	12.91	12.67	12.44	12.21	12.00	11.79	11.59	11.40
20.....	24.00	23.23	22.50	21.82	21.18	20.57	20.00	19.46	18.95	18.46	18.00	17.56	17.14	16.74	16.36	16.00	15.65	15.32	15.00	14.69	14.40	14.12	13.85	13.58	13.33	13.09	12.86	12.63	12.41	12.20	12.00
21.....	25.20	24.39	23.63	22.91	22.24	21.60	21.00	20.43	19.89	19.38	18.90	18.44	18.00	17.58	17.18	16.80	16.43	16.09	15.75	15.43	15.12	14.82	14.54	14.26	14.00	13.75	13.50	13.26	13.03	12.81	12.60
22.....	26.40	25.55	24.75	24.00	23.29	22.63	22.00	21.41	20.84	20.31	19.80	19.32	18.86	18.42	18.00	17.60	17.22	16.85	16.50	16.16	15.84	15.53	15.23	14.94	14.67	14.40	14.14	13.89	13.66	13.42	13.20
23.....	27.60	26.71	25.88	25.09	24.35	23.66	23.00	22.38	21.79	22.23	20.70	20.20	19.71	19.26	18.82	18.40	18.00	17.62	17.25	16.89	16.56	16.24	15.92	15.62	15.33	15.05	14.79	14.53	14.28	14.03	13.80
24.....	28.80	27.87	27.00	26.18	25.41	24.69	24.00	23.35	22.74	22.15	21.60	21.07	20.57	20.09	19.64	19.20	18.78	18.38	18.00	17.63	17.28	16.94	16.62	16.30	16.00	15.71	15.43	15.16	14.90	14.64	14.40
25.....	30.00	29.03	28.13	27.27	26.47	25.71	25.00	24.33	23.68	23.08	22.50	21.95	21.43	20.93	20.45	20.00	19.57	19.15	18.75	18.37	18.00	17.65	17.31	16.98	16.67	16.36	16.07	15.79	15.52	15.25	15.00
26.....	31.20	30.19	29.25	28.36	27.53	26.74	26.00	25.30	24.63	24.00	23.40	22.83	22.29	21.77	21.27	20.80	20.35	19.91	19.50	19.10	18.72	18.35	18.00	17.66	17.33	17.02	16.71	16.42	16.14	15.86	15.60
27.....	32.40	31.35	30.38	29.45	28.59	27.77	27.00	26.27	25.58	24.92	24.30	23.71	23.14	22.60	22.09	21.60	21.13	20.68	20.25	19.84	19.44	19.06	18.69	18.34	18.00	17.67	17.36	17.05	16.76	16.47	16.20
28.....	33.60	32.52	31.50	30.55	29.65	28.80	28.00	27.25	26.53	25.85	25.20	24.59	24.00	23.44	22.91	22.40	21.91	21.45	21.00	20.57	20.16	19.76	19.38	19.02	18.67	18.33	18.00	17.68	17.38	17.08	16.80
29.....	34.80	33.68	32.63	31.64	30.71	29.83	29.00	28.22	27.47	26.77	26.10	25.46	24.86	24.28	23.73	23.20	22.70	22.21	21.75	21.31	20.88	20.47	20.08	19.70	19.33	18.98	18.64	18.32	18.00	17.69	17.40
30.....	36.00	34.84	33.75	32.73	31.76	30.85	30.00	29.19	28.42	27.69	27.00	26.34	25.71	25.12	24.55	24.00	23.48	22.98	22.50	22.04	21.60	21.18	20.77	20.37	20.00	19.64	19.29	18.95	18.62	18.31	18.00
31.....	37.20	36.00	34.88	33.82	32.82	31.89	31.00	30.16	29.37	28.62	27.90	27.22	26.57	25.95	25.36	24.80	24.26	23.74	23.25	22.78	22.32	21.88	21.46	21.06	20.67	20.29	19.93	19.58	19.24	18.92	18.60
32.....	38.40	37.16	36.00	34.91	33.88	32.91	32.00	31.14	30.32	29.54	28.80	28.10	27.43	26.79	26.18	25.60	25.04	24.51	24.00	23.51	23.04	22.59	22.15	21.74	21.33	20.95	20.57	20.21	19.86	19.53	19.20
33.....	39.60	38.32	37.13	36.00	34.94	33.94	33.00	32.11	31.26	30.46	29.70	28.98	28.29	27.63	27.00	26.40	25.83	25.28	24.75	24.24	23.76	23.29	22.85	22.42	22.00	21.60	21.21	20.84	20.48	20.14	19.80
34.....	40.80	39.48	38.25	37.09	36.00	34.97	34.00	33.08	32.21	31.38	30.60	29.85	29.14	28.47	27.82	27.20	26.61	26.04	25.50	24.98	24.48	24.00	23.54	23.09	22.67	22.25	21.86	21.47	21.10	20.75	20.40
35.....	42.00	40.65	39.38	38.18	37.06	36.00	35.00	34.06	33.16	32.31	31.50	30.73	30.00	29.30	28.64	28.00	27.39	26.81	26.25	25.71	25.20	24.71	24.23	23.77	23.33	22.91	22.50	22.11	21.72	21.35	21.00
36.....	43.20	41.81	40.50	39.27	38.12	37.03	36.00	35.03	34.11	33.23	32.40	31.61	30.86	30.14	29.45	28.80	28.17	27.57	27.00	26.45	25.92	25.41	24.92	24.45	24.00	23.56	23.14	22.74	22.34	21.97	21.60

The following is a partial list of mechanical fabrics in general use:

Tire Builder Fabric.—A square woven fabric having usually 11-ply yarn in both warp and filling.

Tire Cord Fabric.—A fabric consisting of hawser cord yarn in the warp, with single yarn filling at intervals to keep warp threads together.

Hose Duck.—A soft, plain-weave fabric of plied yarns not finer than No. 8, made in weights from 10 to 24 oz. to the 40-in. width. When made of finer yarn than No. 8, it is classed as Special Hose Duck.

Rubber Belt Duck.—A soft, plain-weave fabric of plied yarns not finer than No. 8, weight ranging from 22 to 36 oz. to 42-in. width. When made of finer yarn than No. 8, it is classed as Special Belt Duck.

Balata Belt Duck.—A closely woven fabric made of hard twisted plied yarn usually made in wide widths, the weight being based on the square yard.

Oil Belt Duck.—A closely woven fabric of plied yarns not over No. 8, made in a variety of widths, usually a 32-oz. fabric, the weight being based on the square yard.

Numbered Duck.—A plain woven fabric of plied yarns ranging from coarse to fine numbers, the weight being based on 22-in. width.

Army Duck.—A closely woven fabric of plied yarns, usually ranging between 7 to 12 oz., the weight being based on 28½-in. width.

Single Filled or Flat Duck.—A fabric made of single yarn, plain weave, the weight being based on 29-in. width.

Enameling Duck.—A plain woven fabric with laid warp and plied yarn filling, weight being based on 46½-in. width.

(C) *Cotton Fabrics Other than Mechanical Fabrics*

Twill.—A weave which produces pronounced lines running diagonally across the fabric, some of which are known as follows:

Drills.—A commercial term for a fabric with warp face twill usually made with three harnesses and single yarns.

Alberts.—A commercial term for a fabric of twill weave with filling face usually made with four or five harnesses.

Jeans.—A commercial term for a fabric with warp face twill like a drill but with higher count and finer yarns.

Serge.—A commercial term for a fabric of twill weave made with four harnesses 2 by 2.

Clay.—A commercial term for a fabric of twill weave similar to a serge, but made with six harnesses 3 by 3.

(*D*) *General Definitions*

Standard Atmosphere.—In the case of textiles, an air condition in which the relative humidity is 65 per cent at a temperature of 21° C. (70° F.).

Standard Condition.—In the case of textiles, the moisture condition of a material when it is in moisture equilibrium with a standard atmosphere.

Standard Regain.—In the case of any specific textile material, an agreed or officially adopted percentage allowance of moisture added to the absolute dry weight of a material to determine its standard weight.

Standard Weight.—In the case of textiles, the absolute dry weight of a material plus its standard regain.

Moisture Content (*Moisture, Percentage of Moisture*).—In the case of textiles, moisture present in a material shown as a percentage of the net weight.

Yarn.—An assemblage of fibers or filaments of animal, mineral, or vegetable origin, either natural or manufactured, twisted or laid together to form a strand or group of strands for use in weaving, knitting, or forming in any manner into textile fabrics.

Cord.—A string or small rope of fibrous material made by twisting together, generally with hard twist, several yarns for use in tying, binding, lacing, etc., and in weaving or laying very heavy fabrics.

Thread.—A fine cord composed of two or more yarns twisted together, for use in sewing.

Lea.—The division of a hank (840 yd.) of cotton yarn, 120 yd. made up of 80 turns on a standard cotton reel. Also a hank of linen yarn, 300 yd., used as the basis of the linen yarn count.

Count.—In the case of cotton yarn, the number of 840-yd. hanks contained in one pound (avoirdupois 7000 grains) of the yarn at standard condition.

Ends.—The individual warp yarns running lengthwise of a fabric.

Picks.—The individual filling yarns running across a fabric.

Plain Weave.—A fabric pattern in which each yarn of the filling passes alternately over and under a yarn of warp.

Twist, Direction of.—In the case of yarn or cord, the yarn or cord has right-hand or regular twist if when it is held vertically the spirals or twists are seen to incline upward in a right-hand direction and has left-hand or reverse twist when the spirals or twists are seen to incline upward in a left-hand direction.

NOTE.—Attention is called to the fact that this definition of twist is opposite to that used in sewing thread manufacture.

Crimp.—In the case of fabric, the difference in distance between any two points of the yarn as it lies in the fabric, and the same two points after the yarn has been removed from the fabric and straightened, expressed as a percentage of the distance between the two points as the yarn lies in the fabric.

NOTE.—"Crimp" is not to be confused with "take-up" which is the difference in distance between any two points on the yarn as it lies in the fabric and the same two points after the yarn has been removed from the fabric and straightened, expressed as a percentage of the distance between the two points after the yarn has been straightened. The threads for the "take-up" test shall be straightened with the same machine that is used for the "crimp" test, but for this test the machine shall be equipped with an extra scale on the dial, in which case the scale shall be plainly marked "crimp" and "take-up."

Off-Square.—In the case of fabric, the percentage of warp crimp minus the percentage of filling crimp.

STANDARD SPECIFICATIONS
FOR
A.S.T.M. PARTIAL-IMMERSION THERMOMETER FOR GENERAL USE,
−20 TO +150° C., 0 TO +300° F.[1]

Serial Designation: D 182 – 25

These specifications are issued under the fixed designation D 182; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED IN AMENDED FORM, 1925.

1. These specifications cover a partial-immersion thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being −20 to +150° C. or 0 to +300° F., respectively.

2. The purpose of these specifications is to provide a thermometer of sufficient accuracy for general use in determining temperatures as required in tests where no standard thermometer is specified and for adoption as a standard where its design is applicable.

3. The thermometer shall conform to the following requirements:

TYPE: Etched stem, glass.
LIQUID: Mercury.
RANGE AND SUBDIVISION: −20 to +150° C. in 1° C. or 0 to +300° F. in 2° F.
TOTAL LENGTH: 303 to 307 mm. (11.94 to 12.11 in.).
STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6 to 7 mm. (0.236 to 0.275 in.).
BULB: Corning normal or equally suitable thermometric glass.
Length, 19 to 25 mm. (0.75 to 1 in.).
Diameter, 5 to 6 mm. (0.196 to 0.236 in.).
DISTANCE TO −18° C. OR 0° F. LINE FROM BOTTOM OF BULB: 90 to 100 mm. (3.55 to 3.94 in.).
DISTANCE TO 150° C. OR 300° F. LINE FROM TOP OF STEM: 20 to 35 mm. (0.8 to 1.4 in.).
FILLING ABOVE MERCURY: Nitrogen gas.
TOP FINISH: Glass ring.
GRADUATION: All lines, figures and letters clear cut and distinct. The first and each succeeding 5° C. or 10° F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.
IMMERSION: The words "76-mm. immersion" on Centigrade thermometers, or "3-in. immersion" on Fahrenheit thermometers and a line around the stem, 75 to 77 mm. (2.96 to 3.04 in.) above the bottom of the bulb, shall be etched on the thermometer.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-15 on Thermometers.

SPECIAL MARKING: "A.S.T.M.," a serial number and the manufacturer's name or trade mark shall be etched on the stem.

SCALE ERROR: The error at any point of the scale, when the thermometer is standardized as provided below, shall not exceed 0.5° for the Centigrade thermometer or 1° for the Fahrenheit thermometer.

STANDARDIZATION: The thermometer shall be standardized for 76-mm. (3-in.) immersion and for the following temperatures of the emergent mercury column. These stem temperatures have been chosen as corresponding, on the average, to those likely to occur in the use of the thermometer.

THERMOMETER READING	AVERAGE TEMPERATURE OF EMERGENT MERCURY COLUMN
50° C. (122° F.)	35° C. (94° F.)
100° C. (212° F.)	48° C. (118° F.)
150° C. (302° F.)	55° C. (131° F.)

CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. −20 to +150° C., 76-mm. immersion" or "A.S.T.M. 0 to +300° F., 3-in. immersion," according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

STANDARD SPECIFICATIONS
FOR
A.S.T.M. PARTIAL-IMMERSION THERMOMETER FOR GENERAL USE,
−5 TO +300° C., +20 TO +580° F.[1]

Serial Designation: D 183 – 25

These specifications are issued under the fixed designation D 183; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED IN AMENDED FORM, 1925.

1. These specifications cover a partial-immersion thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being −5 to +300° C. or +20 to +580° F., respectively.

2. The purpose of these specifications is to provide a thermometer of sufficient accuracy for general use in determining temperatures as required in tests where no standard thermometer is specified and for adoption as a standard where its design is applicable.

3. The thermometer shall conform to the following requirements:

TYPE: Etched stem, glass.
LIQUID: Mercury.
RANGE AND SUBDIVISION: −5 to +300° C. in 1° C. or +20 to +580° F. in 2° F.
TOTAL LENGTH: 379 to 383 mm. (14.92 to 15.08 in.).
STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6 to 7 mm. (0.236 to 0.275 in.).
BULB: Corning normal or equally suitable thermometric glass.
Length, 10 to 15 mm. (0.39 to 0.59 in.).
Diameter, 5 to 6 mm. (0.196 to 0.236 in.).
DISTANCE TO 0° C. OR 32° F. LINE FROM BOTTOM OF BULB: 90 to 100 mm. (3.94 to 4.33 in.).
DISTANCE TO 300° C. OR 572° F. LINE FROM TOP OF STEM: 25 to 50 mm. (1 to 2 in.).
FILLING ABOVE MERCURY: Nitrogen gas.
TOP FINISH: Glass ring.
GRADUATION: All lines, figures and letters clear cut and distinct. The first and each succeeding 5° C. or 10° F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-15 on Thermometers.

IMMERSION: The words "76-mm. immersion" on Centigrade thermometers, or "3-in. immersion" on Fahrenheit thermometers and a line around the stem, 75 to 77 mm. (2.96 to 3.04 in.) above the bottom of the bulb, shall be etched on the thermometer.

SPECIAL MARKING: "A.S.T.M.," a serial number and the manufacturer's name or trade mark shall be etched on the stem.

SCALE ERROR: The error at any point of the scale, when the thermometer is standardized as provided below, shall not exceed 1° for the Centigrade thermometer, or 2° for the Fahrenheit thermometer.

STANDARDIZATION: The thermometer shall be standardized for 76-mm. (3-in.) immersion and for the following temperatures of the emergent mercury column. These stem temperatures have been chosen as corresponding, on the average, to those likely to occur in the use of the thermometer.

THERMOMETER READING	AVERAGE TEMPERATURE OF EMERGENT MERCURY COLUMN
50° C. (122° F.)	35° C. (94° F.)
100° C. (212° F.)	49° C. (120° F.)
150° C. (302° F.)	61° C. (142° F.)
200° C. (392° F.)	70° C. (158° F.)
250° C. (482° F.)	76° C. (169° F.)
300° C. (572° F.)	80° C. (176° F.)

CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. −5 to +300° C., 76-mm. immersion" or "A.S.T.M. 20 to 580° F., 3-in. immersion," according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

STANDARD SPECIFICATIONS

FOR

A.S.T.M. PARTIAL-IMMERSION THERMOMETER FOR GENERAL USE,

−5 TO +400° C., +20 TO +760° F.[1]

Serial Designation: D 184 − 25

These specifications are issued under the fixed designation D 184; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED IN AMENDED FORM, 1925.

1. These specifications cover a partial-immersion thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being −5 to +400° C. or +20 to +760° F., respectively.

2. The purpose of these specifications is to provide a thermometer of sufficient accuracy for general use in determining temperatures as required in tests where no standard thermometer is specified and for adoption as a standard where its design is applicable.

3. The thermometer shall conform to the following requirements:

TYPE: Etched stem, glass.
LIQUID: Mercury.
RANGE AND SUBDIVISION: −5 to +400° C. in 1° C. or +20 to +760° F. in 2° F.
TOTAL LENGTH: 404 to 408 mm. (15.92 to 16.06 in.).
STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6 to 7 mm. (0.236 to 0.275 in.).
BULB: Corning normal or equally suitable thermometric glass.
Length, 10 to 15 mm. (0.39 to 0.59 in.).
Diameter, 5 to 6 mm. (0.196 to 0.236 in.).
DISTANCE TO 0° C. OR 32° F. LINE FROM BOTTOM OF BULB: 100 to 110 mm. (3.94 to 4.33 in.).
DISTANCE TO 400° C. OR 752° F. LINE FROM TOP OF STEM: 25 to 50 mm. (1 to 2 in.).
FILLING ABOVE MERCURY: Nitrogen gas.
TOP FINISH: Glass ring.
GRADUATION: All lines, figures and letters clear cut and distinct. The first and each succeeding 5° C. or 10° F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.
IMMERSION: The words "76-mm. immersion" on Centigrade thermometers, or "3-in. immersion" on Fahrenheit thermometers and a line around the stem, 75 to 77 mm. (2.96 to 3.04 in.) above the bottom of the bulb, shall be etched on the thermometer.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-15 on Thermometers.

SPECIAL MARKING: "A.S.T.M.," a serial number and the manufacturer's name or trade mark shall be etched on the stem.

SCALE ERROR: The error at any point of the scale, when the thermometer is standardized as provided below, shall not exceed 1° up to 301° C. and 1.5° at higher points in the case of thermometers graduated in Centigrade degrees, or 2° up to 574° F. and 3° at higher points in the case of thermometers graduated in Fahrenheit degrees.

STANDARDIZATION: The thermometer shall be standardized for 76-mm. (3-in.) immersion and for the following temperatures of the emergent mercury column. These stem temperatures have been chosen as corresponding, on the average, to those likely to occur in the use of the thermometer.

THERMOMETER READING	AVERAGE TEMPERATURE OF EMERGENT MERCURY COLUMN
100° C. (212° F.)	50° C. (122° F.)
200° C. (392° F.)	75° C. (167° F.)
300° C. (482° F.)	89° C. (192° F.)
400° C. (572° F.)	94° C. (201° F.)

TEST FOR PERMANENCY OF RANGE: After being subjected to a temperature 10° C. or 20° F. below the maximum scale temperature for 24 hours, the accuracy shall be within the limit specified.

CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. −5 to +400° C., 76-mm. immersion" or "A.S.T.M. 20 to 760° F., 3-in. immersion" according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

STANDARD SPECIFICATIONS
FOR
SIEVES FOR TESTING PURPOSES[1]

Serial Designation: E 11 – 26

These specifications are issued under the fixed designation E 11; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1925; ADOPTED, 1926.

Sieves for precision testing have a variety of uses, including both dry screening and wet screening of material. The primary object is to separate materials according to the size of particle. Since particles are three dimensional and the openings of screens are areas having two dimensions only, the method of separation by means of screens is at best an approximation. In many instances, however, it is the best practical means at hand, and will give useful and reliable results if care is taken in securing representative samples, in the preparation of the sample for testing, in the details of procedure and in the method of reporting results. In the Appendix are indicated those points which must be considered in specifying sieving tests for any individual material.

An essential in each method, however, is a series of sieves of known characteristics. The important consideration in sieves is the size of opening and with the size of opening known it is possible to translate the results obtained in one series of sieves into terms of another series. The importance of the size of opening in the sieve series covered by these specifications is emphasized by designating the sieves in terms of opening in microns. This new scheme of designation should accordingly cause no confusion, giving as it does a direct indication of the size of opening. As such, it is not open to the objections to the various arbitrary number designations heretofore in use and it is also free from the objection to the designation by means of mesh in that it is independent of the size of wire.

With the size of opening known, the method of recording results is simplified. It is recommended that in reporting sieve analyses in graphic form the sizes of sieve opening be arranged as ordinates with the largest size at the top and that the percentages be plotted as abscissas.

Scope.

1. These specifications cover sieves for use in the usual cases of precision testing for the fineness of materials (classification of materials according to size). The sieves have been designated according to the size of openings in terms of microns, the corresponding United States Standard Sieve Series designation in arbitrary numbers being given for the convenience of those familiar with the latter scheme of designation.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

PHYSICAL REQUIREMENTS

Cloth and Frame.

2. Wire cloth for standard sieves shall be woven (not twilled, except that the cloth of 44, 53 and 62-micron sieves may be twilled until further notice) from brass, bronze, or other suitable wire and mounted on the frames without distortion. To prevent the material

TABLE I.—REQUIREMENTS FOR SIEVE OPENINGS AND WIRE DIAMETERS WITH PERMISSIBLE VARIATIONS.

Designation	Corresponding U. S. Standard Sieve Series Number	Sieve Opening		Wire Diameter		Tolerance in Average Opening, per cent	Tolerance on Wire Diameter, per cent		Tolerance in Maximum Opening, per cent
		mm.	in.	mm.	in.		Under	Over	
1	2	3	4	5	6	7	8	9	10
4760 micron.....	4	4.76	0.187	1.27	0.050	±3	15	30	10
4000 "	5	4.00	0.157	1.12	0.044	±3	15	30	10
3360 "	6	3.36	0.132	1.02	0.040	±3	15	30	10
2830 "	7	2.83	0.111	0.92	0.036	±3	15	30	10
2380 "	8	2.38	0.0937	0.84	0.0331	±3	15	30	10
2000 "	10	2.00	0.0787	0.76	0.0299	±3	15	30	10
1680 "	12	1.68	0.0661	0.69	0.0272	±3	15	30	10
1410 "	14	1.41	0.0555	0.61	0.0240	±3	15	30	10
1190 "	16	1.19	0.0469	0.54	0.0213	±3	15	30	10
1000 "	18	1.00	0.0394	0.48	0.0189	±3	15	30	10
840 "	20	0.84	0.0331	0.42	0.0165	±5	15	30	25
710 "	25	0.71	0.0280	0.37	0.0146	±5	15	30	25
590 "	30	0.59	0.0232	0.33	0.0130	±5	15	30	25
500 "	35	0.50	0.0197	0.29	0.0114	±5	15	30	25
420 "	40	0.42	0.0165	0.25	0.0098	±5	15	30	25
350 "	45	0.35	0.0138	0.22	0.0087	±5	15	30	25
297 "	50	0.297	0.0117	0.188	0.0074	±6	15	35	40
250 "	60	0.250	0.0098	0.162	0.0064	±6	15	35	40
210 "	70	0.210	0.0083	0.140	0.0055	±6	15	35	40
177 "	80	0.177	0.0070	0.119	0.0047	±6	15	35	40
149 "	100	0.149	0.0059	0.102	0.0040	±6	15	35	40
125 "	120	0.125	0.0049	0.086	0.0034	±6	15	35	40
105 "	140	0.105	0.0041	0.074	0.0029	±8	15	35	60
88 "	170	0.088	0.0035	0.063	0.0025	±8	15	35	60
74 "	200	0.074	0.0029	0.053	0.0021	±8	15	35	60
62 "	230	0.062	0.0024	0.046	0.0018	±8	15	35	90
53 "	270	0.053	0.0021	0.041	0.0016	±8	15	35	90
44 "	325	0.044	0.0017	0.036	0.0014	±8	15	35	90

NOTE.—Screens having larger openings than those included in the table may be specified, but in specifying screens of larger openings the actual size of the opening (in millimeters or in inches) shall be stated. A tolerance of ±3 per cent on average openings and of 10 per cent in maximum openings shall be permitted.

being sieved from catching in the joint between the cloth and the frame, the joint shall be smoothly filled with solder, or so made that the material will not catch. The sieve frames should be circular, about 20 cm. (8 in.) in diameter and about 5 cm. (2 in.) or 2.5 cm. (1 in.) between the top of the frame and the cloth.

NOTE.—The size specified above is for the regular standard testing sieve, but for special purposes other sizes may be used, as for example, the 3-in. sieve which is designed especially for use on laboratory balances as in the testing of pigments.

Permissible Variations.

3. The average opening between the adjacent warp and the adjacent shoot wires, taken separately, shall be that given in column

3 (or 4) of Table I, within the "Tolerance in Average Opening" given in column 7. The average diameter of the warp and of the shoot wires, taken separately, of the cloth of any given sieve shall be that given in column 5 (or 6) of Table I within the "Tolerance on Wire Diameter" given in columns 8 and 9. The maximum opening between adjacent parallel wires shall not exceed the nominal width of opening for that sieve by more than the "Tolerance in Maximum Opening" given in column 10 of Table I.

WORKMANSHIP

Workmanship.

4. The sieves shall be free from imperfections in the sieve cloth or its mounting, as for example, punctured, loose or wavy cloth, imperfections in soldering, etc.

MARKING

Marking.

5. The sieves shall bear a label upon which the manufacturer's nominal opening in metric units (preferably in microns) with the equivalent in inches, together with the size of wire should be given.

NOTE.—It is recommended that, until the new micron designation shall have been thoroughly established, in addition the corresponding United States Standard Sieve Series number be given.

METHODS OF TESTING

Methods of Testing.

6. (*a*) To determine whether the cloth conforms to the requirements of these specifications, the wire diameter shall be measured in each direction by means of a micrometer-microscope or projection apparatus or other suitable apparatus of requisite precision, care being taken in all cases to avoid diffraction effects in measuring to the edge of a wire. Micrometer calipers and steel scales should not be used for this purpose. The mesh shall be determined by counting the number of openings for a distance of at least 6 in. in both directions of the sieve, using a scale, either glass or steel, and a microscope. The use of scales with special rulings for obtaining mesh by counting "interference bands" is recommended in cases where large numbers of any of the sieves with the smaller openings are to be measured. From the wire diameter and the mesh the average opening shall be computed.

NOTE.—No general rule can be given for the number of observations to be taken, except that they be sufficient to cause no change in the result if say 3 or 4 additional measurements are made.

(*b*) Measurements of large openings shall be made by means of the micrometer-microscope or the projection lantern or other suitable apparatus in the same way as wire diameters are measured.

NOTE.—In order to insure the securing of standard sieves it is possible to obtain from the manufacturer or dealer sieves which have been certified by the U. S. Bureau of Standards.

APPENDIX

LIST OF POINTS TO BE CONSIDERED IN DRAWING UP SPECIFICATIONS FOR A SIEVING TEST

I. APPARATUS.

1. Enumeration of sieves to be used.
2. Auxiliary apparatus required for collection and reduction of gross sample, sample containers, and apparatus other than the sieves used in making the tests.

II. SAMPLING.

1. Unit of sampling.
2. Method of collecting representative gross sample.
3. Size of gross sample.
4. Cautions to be taken in handling sample to prevent contamination or change.
5. Reduction of gross sample.

III. PREPARATION OF THE SAMPLE FOR TESTING.

1. Drying.
2. Mixing.
3. Special treatment, if any is necessary.
4. Selection of test portion.
5. Size of test portion.

IV. PROCEDURE.

A. Dry Screening:

1. Order in which the sieves are to be used.
2. Detailed description of method of shaking.
3. Detailed description of procedure other than method of shaking
4. End point.

B. Wet Screening:

1. Liquid to be used.
2. Description of any preliminary separation by classification or other means.
3. Order in which screens are to be used.
4. Detailed description of procedure including amount and method of adding liquid, method of stirring or other agitation.
5. End point.

V. METHOD OF REPORTING RESULTS.

VI. ALLOWABLE VARIATIONS BETWEEN DUPLICATE TESTS ON THE SAME MATERIAL.

STANDARD METHODS OF VERIFICATION OF TESTING MACHINES[1]

Serial Designation: E 4 – 27

These methods are issued under the fixed designation E 4; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE 1923; ADOPTED IN AMENDED FORM, 1924; REVISED, 1927.

DEFINITIONS

Definitions.

1. (*a*) *Testing Machine.*—A mechanical device for applying a load (force) to a specimen.

NOTE.—Usually the magnitude of the load can be changed at the will of the operator. Many testing machines are arranged to measure the load, but this is not always the case, especially with impact machines and machines for testing ductility.

(*b*) *Error.*—In the case of a testing machine, the value obtained by subtracting the correct value of the quantity measured (usually load) from the indicated value as given by the testing machine.

NOTE.—The error may be positive or negative.

(*c*) *Percentage of Error.*—In the case of a testing machine, the ratio, expressed in per cent, of the error to the correct value of the quantity measured.

(*d*) *Correction.*—In the case of a testing machine, the value obtained by subtracting the indicated value from the correct value of the quantity measured.

NOTE.—The correction has the same magnitude as the error but the opposite sign.

It is recommended that, except for special cases, no corrections be used on machines tested and found to have errors within the tolerances given in these methods.

(*e*) *Tolerance.*—In the case of a testing machine, the maximum allowable error in the value of the quantity indicated.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

NOTE.—It is convenient to express tolerance in terms of percentage of error. The numerical value of the tolerance for a testing machine is so stated hereafter in these methods.

The word "accurate" applied to a testing machine shall be used without numerical values. The word "error" shall be used with numerical values. For example: "An accurate testing machine was used for the investigation"; but "The error of the testing machine at a load of 30,000 lb. was 15 lb." A testing machine is said to be accurate if the indicated load is within the specified tolerance of the actual load.

The *accuracy* of a testing machine should not be confused with *sensitiveness*. For example, a testing machine might be very sensitive—that is, it might indicate quickly and definitely small changes in the load—but, nevertheless, be very inaccurate. On the other hand, the accuracy of the results is, in general, limited by the sensibility.

(*f*) *Loading Range.*—In the case of a testing machine, the range of indicated loads for which the testing machine gives results within the specified tolerances. The loading range shall not include loads either greater than the largest load or less than the smallest load applied in verifying the testing machine.

METHODS FOR VERIFYING TESTING MACHINES THAT MEASURE LOAD

Direct Methods Recommended.

2. Wherever practicable, testing machines shall be verified by applying standard weights directly to the weighing mechanism of the testing machine, or by the use of standardized proving levers in connection with standard weights. The test loads selected shall be applied in both ascending and descending order, and the indications for both the ascending series and the descending series of loads shall be considered in the application of tolerances.

NOTE.—For machines in which the load-indicating device is actuated by a Bourdon pressure tube, a hydraulic (or steam-engine) indicator spring, or other device depending on the elastic properties of a material, the tolerances for the *descending* load shall be applied to differences in the indication for the corresponding ascending and descending readings. In such testing machines the test for ascending load shall be applied as rapidly as practicable after the testing machine has remained unloaded for at least six hours, and the time of applying the ascending series of loads shall not exceed thirty minutes. The descending load test shall be made after the maximum test load has remained on the machine for one hour, and the readings for descending loads shall be taken as rapidly as possible, and in any event the descending load test shall be completed within a period of twenty minutes.

Selection of Test Loads.

3. For any loading range, the testing machine shall be verified by at least four test loads. It is recommended that the test loads

used be 10, 20, 50, and 100 per cent of the maximum capacity at which the testing machine is to be used.

NOTE.—Where a large amount of testing of small specimens is to be done on a large capacity machine, it is recommended that the verification outlined above be supplemented by a series of smaller test loads selected in reference to the light work to be done on the testing machine.

(A) *Method of Verification by Standard Weights*

Method by Standard Weights.

4. Standard metal weights of suitable design, finish, and adjustment shall be placed on the weighing platform of the testing machine or upon trays or other supports suspended from the load-measuring mechanism in place of the specimen. The weights shall be applied

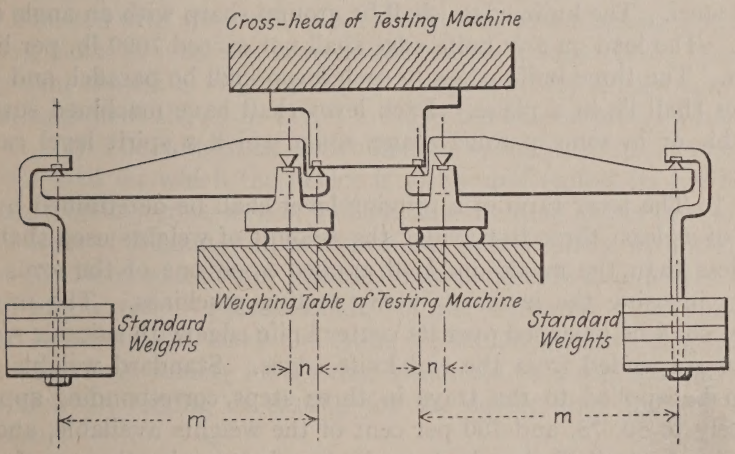

FIG. 1.—Proving Levers.

in increments and removed in the reverse order. They shall be arranged symmetrically with respect to the weighing platform, so that the center of gravity of the load lies in the vertical line through the center of the platform. The applied load and the indicated load shall be recorded for each test load applied, and the error computed from these data.

NOTE.—The method of verification by standard weights can be used only on vertical testing machines. The total load is limited by the size of the platform and the number of weights available. Often twenty weights of 50 lb. each are used, a total of 1000 lb.

This method of verification is seldom used for any but small testing machines, as the loading range does not include the loads for which large machines are used.

(B) Method of Verification by Standardized Proving Levers

Description of Levers.

5. The arrangement of proving levers for verifying testing machines is shown in Fig. 1. The two levers rest on supports on the weighing platform of the testing machine. These supports shall move easily in a horizontal direction, which insures that the forces at each of the knife edges shall be very nearly vertical. The inner knife edges in each lever bear against a suitable block in the movable head of the testing machine. Weight trays or hangers are suspended from each of the outer knife edges and these trays or hangers are loaded with standard weights. The increment of load put on the testing machine by the standard weights is the amount of standard weights multiplied by the lever ratio m/n, Fig. 1.

Specifications of Levers.

6. The knife edges, as well as their supports, shall be of hardened tool steel. The knife edges shall be ground sharp with an angle of 90 deg. The load on any knife edge shall not exceed 7000 lb. per linear inch. The three knife edges in each lever shall be parallel, and their edges shall lie in a plane. Each lever shall have machined surfaces in this or in some parallel plane, upon which a spirit level can be placed.

Verification of Levers.

7. The lever ratio of a proving lever shall be determined by the use of at least three test loads; the amount of weights used shall not be less than the maximum load applied upon one of the arms of a lever in using the levers to verify testing machines. The proving lever shall be balanced over its center knife edge with suitable weight trays suspended from the end knife edges. Standard weights shall then be applied to the trays in three steps, corresponding approximately to 50, 75, and 100 per cent of the weights available, and the proving lever shall then be brought to a balance by the use of small weights and by observations of the freely swinging proving lever. From the weights in the two weight trays the lever ratio shall be computed.

Method of Procedure.

8. The proving levers shall be placed symmetrically in the testing machine to be verified and both levers brought as near to a horizontal position as is feasible, after applying each increment of load, by means of the movable head of the testing machine. The testing machine shall be balanced with the levers in place and the weight trays empty. Standard weights shall be applied in increments, half an increment in each tray, and they shall be removed in the reverse order. The weights shall be placed symmetrically on the weight trays, with the center of gravity of the weights over the center of the tray. The applied load and the indicated load shall

be recorded for each test load applied, and the error computed from these data.

NOTE.—The method of verification by standardized proving levers is used, at the present time, only on vertical testing machines. It is often used up to loads of 10,000 lb.

(C) Method of Verification by Means of an Elastic Calibration Device

Method by Calibration Device.

9. In this method the testing machine to be verified is tested by comparing its load indications with the amount of elastic deformation of a calibration device, the deformation being measured with a delicate strainometer.

Calibration Device.

10. The calibration device may be a bar tested in tension or in compression or an elastic loop or other shape whose elastic deformation can be measured, and if a tension or compression bar is used it shall be fitted with spherical-seated shackles or with a spherical-seated bearing block to insure as nearly axial loading as is possible. The arrangement shown in Fig. 2 or Fig. 3 is suggested for tension bars and the arrangement shown in Fig. 4 is suggested for compression bars.

Adjustment of Calibration Device.

11. (*a*) The calibration device shall be of such size that under the greatest load for which the device is to be used (called its full load) the stress on any cross-section will not be more than 80 per cent of the elastic limit of the material, as determined in accordance with Method II for the Determination of Elastic Limit as described under the definition of the term "Elastic Limit" of the Tentative Definitions of Terms Relating to Methods of Testing (Serial Designation: E 6 – 25 T) of the American Society for Testing Materials[1] on a test specimen cut from the bar, forging or casting for the device after its final heat treatment.

(*b*) Before the calibration device is used for verification purposes, it shall be loaded to its full load at least six times, and between successive loadings it shall be heated to a temperature of 100° C. (212° F.) for a period of thirty minutes or shall be allowed to rest for a period of 24 hours. The object of this treatment is to adjust the device to its bearings and to relieve internal strain.

Loading Range

12. The *loading range* of a calibration device is the range of indicated loads for which the device gives results within specified tolerances. Obviously, the loading range may not include loads either greater than the largest load or less than the smallest load applied in verifying the calibration device.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part I, p. 879 (1925); also 1927 Book of A.S.T.M. Tentative Standards, p. 744.

Method of Procedure.

13. (*a*) The loading range of a calibration device shall be determined by verifying its indications by the use of standard weights or by standard weights and proving levers. In verifying the indications of the device, an initial load of 1 per cent of its full load shall first be applied. All changes of strainometer reading and of applied load shall be reckoned from the readings at this initial load. Successive loads shall be applied, a recommended series of loads being 10, 20, 50

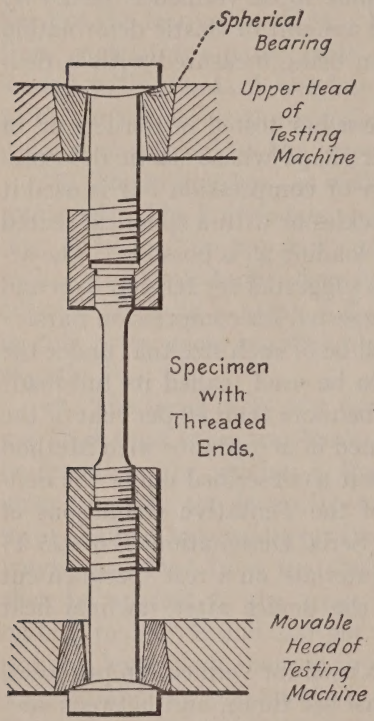

FIG. 2.

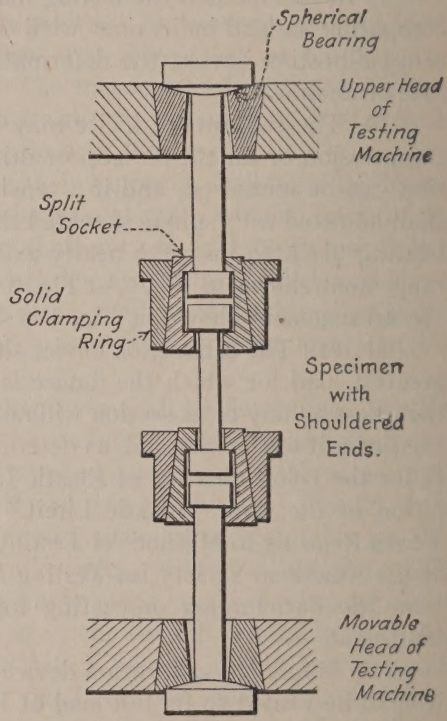

FIG. 3.

and 100 per cent of the full load of the device. Such a series shall be applied to the bar at least five times to determine the relation of applied load to strainometer reading, the shackles or bearing blocks being readjusted before each application of the series.

(*b*) The average of the five (or more) strainometer readings obtained in this way shall be taken for each load applied. The average of the differences (taken without regard to algebraic sign) between the individual strainometer readings for any load and the mean reading for that load constitutes the average deviation for that load. The lowest value of load for which the average deviation is not greater

than 0.5 per cent of the strain for that load is the lower limit of the loading range of the calibration device.

NOTE.—If in taking five or more strainometer readings for any load the deviation of any one reading is more than 1 per cent from the mean reading, then this series of five or more readings shall be repeated.

Strainometer.

14. The strainometer for elastic calibration devices which employ elastic bars in axial tension or axial compression shall be so designed as to indicate the average elongation or compression of the bar. For any elastic calibration device, it shall be sufficiently sensitive to indicate a change of 0.25 per cent of the minimum load in the loading range of the device. If the strainometer is not permanently attached to the calibration device, the strainometer shall be removed and replaced

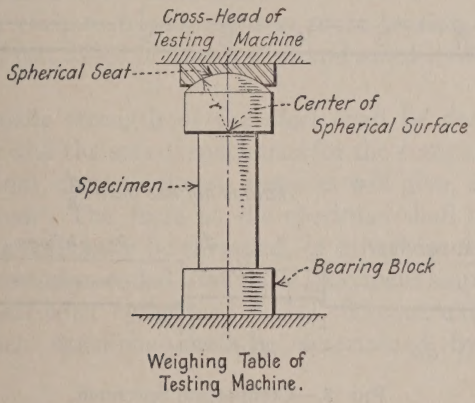

FIG. 4.

for each successive reading taken during the calibration of the device itself. This, of course, does not mean that the strainometer must be removed for each reading in the regular use of the instrument.

Precautions.

15. In using the calibration device it is important that just previous to taking any reading, including the initial reading of a series, there shall be no reversal in the direction of motion of the indicating mechanism of the strainometer, because such reversal may introduce error due to backlash in the instrument. That is, when taking readings for a series of increasing loads the load should be brought up to each predetermined test load, and when taking readings for a series of decreasing loads the load should be brought down to each predetermined test load.

Temperature Correction.

16. In using a calibration device made of steel, correction for variation in mean temperature during any calibrations made on different occasions should be made by allowing a diminution of 0.015

per cent ($\frac{15}{1000}$ of 1 per cent) in the modulus of elasticity of steel for each 1° F. increase in temperature. During any calibration with an elastic calibration device, temperature conditions should be kept as uniform as is possible.

(*D*) *Verification by Comparison Method*

Description of Comparison Method.

17. When the direct method of verification of a testing machine cannot be carried out (as is the case at present with horizontal testing machines, for example), or when an elastic calibration device is not available a comparison method of verification may be used. In this method the machine to be verified is tested by comparing its indications with the corresponding readings obtained under the same con-

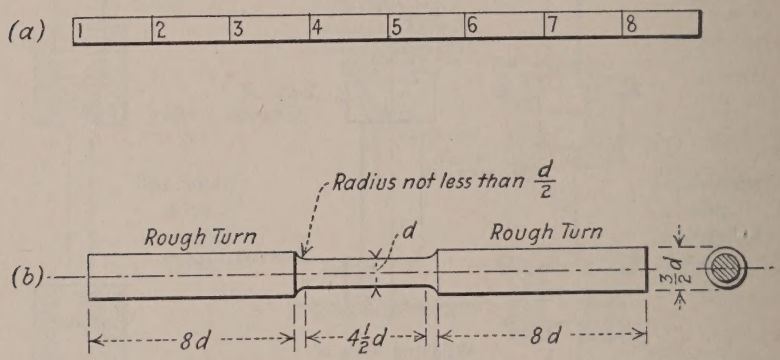

FIG. 5.—Comparison Specimen.
(*a*) Method of Numbering Specimens.
(*b*) Form of Test Specimen.

ditions by the use of another testing machine, especially tested and verified, as specified in Section 18. The method of verification by comparison shall be carried out by the use of a series of companion specimens, half of which are to be tested in tension in the machine to be verified, and half of which are to be tested in tension in the especially verified testing machine which serves as a standard machine. The general provisions respecting the application of the test loads and the loading range which are outlined in the direct method of verifying testing machines shall apply to the verification of testing machines by the comparison method, so far as is practicable.

NOTE.—The method of verification of a testing machine by standard weights (Section 4) and the method of verification by standardized proving levers (Sections 5 to 8) are both regarded as superior to the method of verification by the use of companion specimens. If the results of a verification test of a testing machine by the comparison method fail to agree with the results of a verification by either the

standard-weight method or the proving-lever method, the results of the verification by the comparison method shall be discarded.

Requirements for Testing Machines used for Verifying Other Machines.

18. (*a*) A testing machine which is to be used as the standard for verifying other testing machines shall itself be verified by a special test, in which the verification is carried out twice. The loading range shall be determined by the tolerance for new testing machines, namely, ±1 per cent (Section 22). For the loading range established, the differences in indications found in the two tests of the standard machine shall not exceed 0.25 per cent.

(*b*) In verifying testing machines by the comparison method, corrections shall be applied to the indications of the standard machine and these corrections shall be based on the average correction obtained in the two tests used in its verification.

Test Specimens.

19. (*a*) For each test load, eight or more tension test specimens shall be cut from soft rolled or drawn steel and numbered consecutively, as in Fig. 5 (*a*).

(*b*) The tensile strength of the steel shall be determined by a preliminary test and the sets of specimens for the comparison test shall have such nominal cross-sectional areas as will give, approximately, the loads required. The form of the specimen shall be as shown in Fig. 5 (*b*). The ends may be threaded, or otherwise machined, to fit holders, but it is recommended that those specimens shipped to another laboratory be left with cylindrical ends. The actual value of "*d*" (Fig. 5) for each specimen shall be determined by means of a micrometer.

NOTE.—It should be noted that the standard tension test specimen,[1] 0.5 in. in diameter and having a 2-in. gage length, satisfies the requirements for a specimen for the verification of a testing machine by the comparison method.

Method of Procedure.

20. (*a*) The specimens having odd numbers shall be tested in the machine to be verified and those having even numbers in the standard testing machine. The tensile strength only shall be determined.

(*b*) The speed of the testing machine, by which is meant the speed of the pulling head when the machine is running idle with no specimen in the machine, when testing verification specimens shall be approximately the same for both the standard testing machine and the machine being verified. The speed shall be the slowest speed that can be obtained on both machines; in any case the speed shall not exceed 1 in. per minute per inch of diameter of comparison specimen.

(*c*) When testing verification specimens, especial care shall be

[1] See Part I, 1927 Book of A.S.T.M. Standards, p. 32, Fig. 1.

taken to maintain the weighing beam of the testing machine in equilibrium as the tensile strength of the specimen is approached. It is recommended that a mark be placed at the middle of the trig loop and that a pointer be placed on the beam for verification tests, and that the pointer be kept as nearly on the mark as possible.

Computation of Error.

21. (*a*) The average tensile strength (in pounds per square inch) of the odd-numbered specimens, multiplied by the nominal area of cross-section, shall be considered a measure of the indicated load, and the average tensile strength of the even-numbered specimens, multiplied by the nominal area of cross-section, shall be considered a measure of the applied load. The error shall be computed from these data.

(*b*) If the tensile strength (in pounds per square inch) of any verification specimen varies more than 1 per cent from the mean of the group with which it is tested, its tensile strength shall not be used in computing the error of the testing machine. If more than one specimen in the odd-numbered group or one in the even-numbered group exceeds the allowable variation in tensile strength, all the specimens shall be discarded and another set prepared.

TOLERANCES FOR TESTING MACHINES THAT MEASURE LOAD

Tolerances.

22. All testing machines that measure load shall conform to the following tolerances:[1]

(*a*) *New Machines.*—The error for loads in the loading range shall not exceed ±1.0 per cent for new machines or for those which have had any of the knife edges in the weighing mechanism repaired or renewed.

(*b*) *Used Machines.*—The error for loads in the loading range shall not exceed ±1.5 per cent.

Corrections.

23. The indicated load of a testing machine shall not be corrected either by computation or by the use of a calibration diagram to obtain values within the required tolerance.

TIME INTERVAL BETWEEN VERIFICATIONS

Time Interval Between Verifications.

24. It is recommended that testing machines, when in constant use, be verified at intervals of six months and, when used intermittently, at intervals of two or three years. Testing machines shall, however, be verified immediately after making repairs or adjust-

[1] It is believed that the tolerances given in this section may be made much smaller when more perfect methods of verification become generally available; and that the tolerances might well be materially reduced if there is available equipment for verifying testing machines by the use of standard weights (Section 4) or by the use of standardized proving levers (Sections 5 to 8).

ments of the weighing mechanism, and whenever there is reason to doubt the accuracy of the results, without regard to the time interval since the last verification.

REPORTS AND CERTIFICATES

Reports.

25. A clear and complete report shall be prepared of each verification of a testing machine. This report shall contain all data necessary to verify the accuracy of the report, and shall be filed for future reference. This report shall state clearly the method of verification used.

Certificates.

26. A certificate giving the maker's serial number and a brief description of the testing machine, the maker's name, the date of verification, and the loading range, shall be signed by the person responsible for the maintenance of the testing machine, and this certificate shall be posted in plain view of a person operating the testing machine.

STANDARD DEFINITIONS OF TERMS RELATING TO SPECIFIC GRAVITY[1]

Serial Designation: E 12 – 27

These definitions are issued under the fixed designation E 12; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1925; ADOPTED IN AMENDED FORM, 1927.

1. *Absolute Specific Gravity* (of solids and liquids).—The ratio of the weight referred to vacuum of a given volume of the material at a stated temperature to the weight referred to vacuum of an equal volume of gas-free distilled water[2] at a stated temperature. It shall be stated thus:

(*a*) When the temperatures of the material and of the water are the *same:*

Absolute Specific Gravity $x°$ C., ...,

where x is the temperature of the material and the water.

(*b*) When the temperatures of the material and of the water are *not the same:*

Absolute Specific Gravity $\frac{x° \text{ C.}}{y° \text{ C.}}$ .....,

where x is the temperature of the material and y the temperature of the water.

NOTE.—In the interest of standardization and simplification, the first form of statement of specific gravity (*a*) should be employed wherever practicable.

2. *Specific Gravity* (of solids and liquids).—The ratio of the weight in air of a given volume of the material at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

(*a*) When the temperatures of the material and of the water are the *same:*

Specific Gravity $x°$ C., ...,

where x is the temperature of the material and the water.

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee E-8 on Nomenclature and Definitions.

[2] Distilled water boiled vigorously in vacuum.

(*b*) When the temperatures of the material and of the water are *not the same:*

Specific Gravity $\frac{x° \text{ C.}}{y° \text{ C.}}$ .....,

where x is the temperature of the material and y is the temperature of the water.

NOTE.—In the interest of standardization and simplification, the first form of statement of specific gravity (*a*) should be employed wherever practicable.

3. *Apparent Specific Gravity* (of solids).—The ratio of the weight in air of a given volume of the impermeable portion of a permeable material (that is, the solid matter including its impermeable pores or voids) at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

(*a*) When the temperatures of the material and of the water are the *same:*

Apparent Specific Gravity $x°$ C., ...,

where x is the temperature of the material and the water.

(*b*) When the temperatures of the material and of the water are *not the same:*

Apparent Specific Gravity $\frac{x° \text{ C.}}{y° \text{ C.}}$ .....,

where x is the temperature of the material and y is the temperature of the water.

NOTE 1.—In scientific circles, specific gravity and density determinations made in air (that is, uncorrected to vacuum) are frequently distinguished by the adjective "apparent." Thus, the specific gravity defined by definition No. 2 would be designated as "apparent specific gravity" and that defined by definition No. 1 as "specific gravity." But in industry, the terminology is more generally in accordance with that given in these definitions.

NOTE 2.—The terms "permeable" and "impermeable" cannot be rigidly defined for general application. The exact meaning in a particular application is the conventional one inferred by the procedure specified for determining the specific gravity of the material in question.

NOTE 3.—In the interest of standardization and simplification, the first form of statement of specific gravity (*a*) should be employed wherever practicable.

4. *Bulk Specific Gravity* (of solids).—The ratio of the weight in air of a given volume of a permeable material (including both permeable and impermeable voids normal to the material) at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

(*a*) When the temperatures of the material and of the water are the *same:*

Bulk Specific Gravity $x°$ C., ...,

where x is the temperature of the material and the water.

(*b*) When the temperatures of the material and of the water are *not the same:*

Bulk Specific Gravity $\frac{x° \text{ C.}}{y° \text{ C.}}$ .....,

where x is the temperature of the material and y is the temperature of the water.

NOTE 1.—See Note 2 under "Apparent Specific Gravity."

NOTE 2.—In the interest of standardization and simplification, the first form of statement of specific gravity (*a*) should be employed wherever practicable.

REGULATIONS GOVERNING STANDING COMMITTEES

ADOPTED, 1910; LATEST REVISION, 1927.

NOTE.—By action of the Executive Committee on January 6, 1912, the responsibility for the general Regulations Governing Standing Committees is vested in the Executive Committee and Committee E-5 on Standing Committees, with the understanding (1) that a proposed change in these Regulations originating with Committee E-5 shall be subject to approval by the Executive Committee of the Society; (2) that the Executive Committee of the Society shall make no changes in these Regulations without first referring the same to Committee E-5; and (3) that proposed changes in these Regulations thus adopted shall be announced in the next circular to members and become effective from the date of issue of that circular.

I. ORGANIZATION OF STANDING COMMITTEES

1. Creation.—The creation of a standing committee shall be subject to the authorization of the Executive Committee, acting either on a recommendation adopted by majority vote at an annual meeting of the Society, or on its own initiative.

2. Appointments and Classifications.—Appointments on standing committees shall be made by the Executive Committee subject to the following provisions:

(*a*) On committees dealing with subjects having a commercial bearing, the members of the committee shall be classified into "producers," "consumers," and "general interests," defined as follows:

Producers.—Those directly concerned in the production and sale of the commodity involved;

Consumers.—Those that use the commodity involved but are not directly concerned with its production and sale;

General Interests.—Independent engineers, educators and persons who are neither consumers nor producers as defined above.

On such committees either an equal numeric balance shall be maintained between the producers on the one hand, and the consumers and general interests, jointly, on the other hand; or the consumers and general interests may be allowed to predominate by majority vote of the producers.

(*b*) The classification of the members of a committee into producers, consumers, and general interests shall be left to each committee, subject to the following provisions, and with the understanding that a member dissatisfied with this classification has the right of appeal to the Executive Committee:

(1) A member who stands in the relation of producer to any product within the province of the committee shall be classed as a producer, although at the request of the officers of the committee concerned,

attention shall be called to the status of such members in a footnote worded as follows:

These members of Committee....., classed as Producers, stand in the relation of Producers to certain products, and in that of Consumers (or General Interests) to other products within the province of the committee.

(2) A nominally unattached expert, who is permanently retained by producing interests in the field of activities of the committee with which he is connected, shall be classed as a producer. The qualification "permanently retained" is to be understood to mean that the expert receives a regular monthly or yearly retainer from one or more producing interests under an indefinitely continuing arrangement.

(*c*) As a general policy, only one individual connected with a given firm, company, corporation, laboratory, or other institution shall be eligible to membership on a given committee, although exceptions to this rule may be permitted at the discretion of the committee concerned. In case two or more members of a committee are connected with the same firm, company, corporation, laboratory, or other institution, they shall, when a division is demanded, jointly command only a single vote.

(*d*) Additional appointments on existing committees shall be made only on the recommendation of, or with the approval of, such committees.

(*e*) Only members of the Society shall be eligible, in general, to appointment on committees, although exceptions may be authorized by the Executive Committee in favor of representatives of government branches or other societies.

3. Preliminary Organization.—The President of the Society will appoint the chairman *pro tem.* of a new committee from among the consumers or general interests. The chairman *pro tem.*, after communicating with the other members of the committee, will fix the place and time of the first meeting. He may, at his discretion, appoint one or more members of the committee to prepare material in advance for consideration at that meeting or he may prepare such material himself. This procedure is recommended as calculated to economize time at the meeting and to afford a definite basis for discussion.

4. Permanent Organization.—At the first meeting of a committee a permanent organization shall be effected by the election of a permanent chairman from among the consumers or general interests, and such other officers as the committee may desire, and the creation of such sub-committees as may be desired. The duties and powers assigned to these officers and sub-committees, and the details of management and administration in general, shall be at the discretion of each committee, subject to the limitations of these Regulations.

II. DIRECTIONS FOR CONDUCT OF BUSINESS

5. Meetings.—The meetings of standing committees and sub-committees shall be open only to their own members and to such visitors whose proposed invitation has been approved by the chairman.

Minutes of meetings of standing committees shall be kept and copies thereof shall regularly be sent to the Secretary-Treasurer of the Society.

6. Proxies.—A member of a standing committee shall be authorized to delegate any desired individual as his proxy with voting power, or without voting power if so specified; but no individual shall have more than one vote at a meeting of a committee.

7. Election of Officers.—Every standing committee shall hold an election of officers at or before the annual meeting of the Society occurring in the even years. The result of such election shall be included in the report of the committee for that year. The term of office of every officer shall be two years and officers shall be eligible for re-election.

8. Resignations.—Proposed resignations from office or from membership on a standing committee shall be reported directly to the chairman or the secretary of the committee concerned, and the result of any action taken in such matters shall be reported to the Secretary-Treasurer of the Society.

9. Sub-Committees.—Sub-committees shall have no standing in the Society except through their parent committees. Sub-committees on proposed complete standard specifications for materials shall consist of not fewer than six members, and at least one-half of the membership shall be composed of consumers and general interests. Departures from this requirement for exceptional reasons may be authorized by the Executive Committee.

III. STANDARDS AND RECOMMENDED PRACTICE

10. Standards.—"The term 'Standards' shall be applied collectively to (*a*) standard specifications, (*b*) standard methods, and (*c*) standard definitions."[1]

(*a*) The term "Standard Specifications" shall be applied to specifications designed to govern the purchase of materials. Such specifications may or may not include reference to tests, but they shall include limits for physical, chemical or other properties.

(*b*) The term "Standard Methods" shall be applied to methods of testing, analysis, sampling and similar operations. Standard Methods shall not include limits for physical, chemical or other properties.

(*c*) The term "Standard Definitions" is self-explanatory.

11. Tentative Standards.—The term "Tentative Standards" shall be applied to proposed standards which are published in the Proceedings for one or more years with a view of eliciting criticism, of which the committee concerned will take due cognizance before recommending final action toward their adoption as standard.

12. Recommended Practice.—"The term 'Recommended Practice' shall be applied to processes and methods not ordinarily subject to contract between purchaser and manufacturer."[1]

13. Matters of Engineering Design.—In the preparation of proposed standards the consideration of matters of engineering design or construction shall not in general be regarded as falling within the province of the Society. If, however, it should appear to a given committee that the consideration of

[1] Quoted from By-laws, Article VI, Section 1.

such matters is, for special reasons, indispensable in specifications designed to cover the customary relations between the producers and consumers of a given product, then reference to such matter in proposed specifications for that product shall be permitted within the scope necessary for the particular purpose above stated. Proposed standards embodying features of the character in question shall be submitted by the committee concerned to the Executive Committee for consideration and comment not later than the quarterly meeting immediately preceding the annual meeting at which the proposed standards are to be presented.

14. Preparation of Standards.—Proposed new standards or proposed amendments of existing standards shall originate in the particular committee within whose province such standards properly belong. No action affecting standards shall be taken by any standing committee except at meetings called for that purpose. Action at such meetings shall be subject to a two-thirds vote of those voting, and subsequently to a two-thirds vote of those voting on letter ballot of the entire committee. The results of each letter ballot as to the number of affirmative votes, the number of negative votes, and the number of members not voting, shall be announced in the report of the committee to the Society.

Dissenting members shall have the right to present minority reports, individually or jointly, at the annual meeting of the Society at which the majority report is presented.

15. Procedure Governing the Adoption of Standards.—Any recommendations affecting standards presented by the appropriate committees at the annual meeting of the Society shall be subject to the following provisions in Article VI, Section 1, of the By-laws:

"Proposed new standards shall be presented at an annual meeting, at which they may be amended by a two-thirds vote of those voting. On two-thirds vote of those voting, they shall be printed, as presented or as amended, in the Proceedings and separately under the title 'Tentative Standards,' on which written discussions addressed to the appropriate committee shall be invited. At the next annual meeting tentative standards shall be subject to amendment by a two-thirds vote of those voting. They may then be referred, by a like vote, to letter ballot of the Society, in which case a two-thirds vote of those voting shall be required for adoption; or, on the recommendation of the committee concerned, they may be continued as tentative, as printed or as amended, in which case the above prescribed procedures shall apply at any succeeding annual meeting.

"Proposed amendments of existing standards shall be presented at an annual meeting, at which they may be amended by a two-thirds vote of those voting. On two-thirds vote of those voting they shall be printed, as presented or as amended, in the Proceedings as part of the report of the appropriate committee; and collectively under the same cover with the Tentative Standards for that year. At any succeeding annual meeting they shall be subject to amendment by a two-thirds vote of those voting; and at the annual meeting in the year in which the book of A.S.T.M. Standards will next be published, they shall be subject to reference by a like vote to letter ballot of the Society, in which case a two-thirds vote of those voting shall be required for adoption.[1]

[1] *Interpretation.*—The Executive Committee and Committee E-5 on Standing Committees have ruled that proposed revisions of amendments that have previously been accepted for publication as tentative must be germane to the original amendment in order to come under this provision. Revisions that are not germane to the original amendments shall be considered as new amendments.

"The above requirement by which final action on proposed new standards or proposed amendments of existing standards shall be deferred for one or more years may, for exceptional reasons, be waived by a nine-tenths vote of those voting at the annual meeting at which they are first presented. In that case the above prescribed vote as to amendments, as to reference to letter ballot, and as to adoption shall remain unaffected."

"The above requirements governing action on proposed new standards or proposed amendments of existing standards shall be applicable also to proposed Recommended Practice."

16. Standards Involving Patents.—"Reports, resolutions and recommendations pertaining to or involving the use, or proposed use, in a standard or tentative standard, of any device or process which forms the subject matter of any existing patent, shall first be submitted to the Executive Committee, and shall be submitted to the Society only with the approval of the Executive Committee."[1]

Rulings.—The Executive Committee has adopted the following policies as applicable to standards involving patents:

Applying to the specifying of patented apparatus in A.S.T.M. Standards:

Where a standing committee desires to include in a standard of the Society the use of a patented instrument, appliance or machine for testing, for which the committee has written certain specifications, and where this instrument, appliance or machine is the only one specified in the standard, the Executive Committee will approve its inclusion in the standard provided the owner of the patent will execute a satisfactory agreement with the Society to license any reputable manufacturer approved by the Executive Committee to manufacture such instruments, appliances or machines for testing in accordance with the specifications of the Society.

Where the patented instrument, appliance or machine concerned is one of several alternative instruments that may be used in applying the standard of the Society, the other instruments not being patented, the Executive Committee will permit reference to such patented instrument without requiring the execution of an agreement such as above outlined, in the belief that the interests of the user of the standard are fully protected against an undesirable patent monopoly in that he can use other unpatented instruments in accordance with the provisions of the standard.

Applying to standards involving patented materials or patented processes:

The Executive Committee will consider upon their respective merits any proposals from the standing committees of the Society that specifications be prepared involving patented materials or patented processes. If such specifications are adopted by the Society, the Society does not undertake to insure any one utilizing such specifications against liability for infringement of any patent or assume any such liability, and such adoption does not constitute a recommendation of any patented or proprietary application that may be involved.

IV. REPORTS OF STANDING COMMITTEES

17. Reports.—The reports of standing committees shall be presented at the annual meetings. The report of every sub-committee shall be made to the parent committee and not to the Society direct. If such a report is embodied

[1] Quoted from By-laws, Article VI, Section 2.

wholly or in part in the report of the parent committee to the Society, the latter shall make definite references to such features in its own report and recommendations, if any, based thereon.

Standing committees shall include in their annual reports, in concise form, a statement of the essential facts regarding the origin of all proposed new standards and the reasons for proposing amendments in existing standards and tentative standards. Appropriate data respecting new methods of test or proposed modifications of existing methods of test shall be included in such annual reports or filed with Committee E-1 on Methods of Testing, as may seem preferable. Reference to available publications shall be accepted as appropriate data.

Standing committees shall include in their annual reports, or in a formal statement to the Executive Committee of the Society in case there is no report for a given year, their reasons for continuing a tentative standard or tentative revision of an existing standard in the tentative status for more than three years without revision or without recommendation for advancement to standard.

The results of letter ballot votes of the committee on actions affecting standards shall be announced in the report of the committee (see Section 14).

The report of a standing committee, before its presentation at the annual meeting, shall first have been submitted to letter ballot of the committee and shall have received the approval of at least two-thirds of those voting.

A statement of the following form shall appear at the close of every committee report:

> This report has been submitted to letter ballot of the committee which consists of ..... members; ..... members returned their ballots, of whom ..... have voted affirmatively, ... negatively, and ..... have marked their ballots "not voting."

Dissenting members shall have the right to present minority reports individually or jointly.

18. Advance Distribution of Reports.—The annual reports of the standing committees shall be transmitted to the Secretary-Treasurer of the Society as early in the calendar year as possible and not less than eight weeks in advance of the date of the annual meeting. Preprints of these reports shall be mailed by the Secretary-Treasurer to every member of the Society who has signified his desire to receive such reports, at the earliest possible subsequent date and not less than four weeks before the annual meeting, so that members may come to the meeting prepared to discuss such reports and that members not intending to be present at the meeting may contribute discussions by letter.

V. COOPERATION WITH COMMITTEES OF OTHER BODIES

19. Methods of Initiating Cooperation.—A standing committee desiring to cooperate with committees of other bodies on like or cognate subjects, or to bring about the appointment of similar committees by other bodies for purposes of cooperation, shall address a recommendation to that effect to the Executive Committee and, on the approval of the latter, negotiations to the desired end shall be conducted by the Secretary-Treasurer of the Society. Such cooperative relations shall entail no conditions at variance with these Regulations, and shall impose no restrictions upon the free and independent action of the standing committee.

20. Regulations Governing Cooperative Relations.

While ample opportunity is afforded for the standing committees of the Society to cooperate with other organizations through the formation of joint committees under suitable regulations, the whole question of cooperative work between societies has been in such a state of flux during recent years that it has been deemed unwise to endeavor to supply the subject matter of Section 20 of these Regulations.

Accordingly this section is temporarily left blank in anticipation of supplying its contents later.

VI. PUBLICATIONS

21. Publications.—Committees shall have no right to issue matter for publication through other than the regular Society channels, unless so authorized, for exceptional reasons, by the Executive Committee.

VII. EXPENSES OF STANDING COMMITTEES

22. Current Expenses.—Expenses for postage incurred in connection with the business of committees will be refunded by the Secretary-Treasurer of the Society on vouchers approved by the chairmen of these committees.

23. Stationery.—Correspondence relating to the business of committees or sub-committees shall be conducted on official stationery which will be furnished by the Secretary-Treasurer of the Society.

24. Extraordinary Expenses.—Expenses for items other than postage will not be assumed by the Society, unless such expenditures were incurred in pursuance of previous authorization of the Executive Committee, on recommendation of the chairman of the committee concerned, and within amounts specifically fixed by the Executive Committee.

25. Special Funds.—Committees engaged on subjects having a commercial bearing shall be authorized to solicit contributions from manufacturers towards research funds. Contributions from consumers to funds for this and other purposes shall be solicited only by the Executive Committee. All funds thus collected shall be transmitted to the Secretary-Treasurer of the Society and deposited by him in bank and placed to the credit of the committees on the books of the Society, subject to disbursement only on voucher signed by the chairman of the committee concerned.

26. Salaries and Fees.—Committees shall not be authorized to pay salaries or professional fees in any form to any of their officers or members. Assistants in connection with research work may be engaged at salaries or special compensation fixed by the committees concerned, provided that funds for such salaries or compensations shall previously have been deposited with the Secretary-Treasurer of the Society. Payments for such purposes shall be made by the Secretary-Treasurer of the Society only on vouchers approved by the chairman of the committee concerned.

VIII. DISCHARGE OF STANDING COMMITTEES

27. Discharge of Standing Committees.—Standing committees may be discharged by the Executive Committee, either at their own request or with their consent, on the completion of the work for which they were appointed, or in consequence of protracted inactivity. A standing committee which fails to present a report at three successive annual meetings of the Society will be required to show cause, in a written communication to the Executive Committee, why it should not be discharged.

Standing committees may be discharged for cause by the Executive Committee at its own initiative.

IX. METHODS AND INSTRUMENTS OF TESTING

28. Methods and Instruments of Testing.—Regulations Governing Methods and Instruments of Testing, adopted jointly by Committee E-5 on Standing Committees and the Executive Committee and made effective June 22, 1921, form part of the Regulations Governing Standing Committees. These regulations establish the responsibility of the standing committees for the preparation of methods of testing and the examination, design and recommendation of instruments for testing, as well as the authority of Committee E-1 on Methods of Testing and the relation between the standing committees and Committee E-1.[1]

X. NOMENCLATURE AND DEFINITIONS

29. Nomenclature and Definitions.—Regulations Governing Nomenclature and Definitions, adopted jointly by Committee E-5 on Standing Committees and the Executive Committee and made effective June 22, 1921, form part of the Regulations Governing Standing Committees. These regulations establish the responsibility of the standing committees for the preparation of nomenclature and definitions, as well as the authority of Committee E-8 on Nomenclature and Definitions and the relation between the standing committees and Committee E-8.[2]

COOPERATIVE POSSIBILITIES BETWEEN U. S. GOVERNMENT BRANCHES AND A.S.T.M. STANDING COMMITTEES

The basis on which various government branches may cooperate with the committees of this Society is indicated in the 1927 Year Book, pages 333–334. Any committee desiring to establish such cooperative relations shall address a recommendation to that effect to the Executive Committee, and on approval by the latter, negotiations to the desired end shall be conducted by the Secretary-Treasurer of the Society.

SUPPLEMENTARY REGULATIONS GOVERNING STANDING COMMITTEES

The standing committees of the Society may adopt supplementary regulations governing their various activities. Such regulations have been adopted by a number of committees of the Society.

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[1] These regulations appear in the 1927 Year Book, p. 325.

[2] These regulations appear in the 1927 Year Book, p. 329.

LIST OF A.S.T.M. STANDARDS

As of September 1, 1927. Total Number, 340.

Published collectively in their latest revised form in 1927 BOOK OF A.S.T.M. STANDARDS—Parts I and II

These Standards are all copyrighted in the name of the American Society for Testing Materials. Permission to reprint any of these Standards can be obtained only from the Executive Committee on application to the Secretary-Treasurer.

The designations A 1, A 2, etc., of the Standards are fixed; the final numbers 14, 24, etc., indicate the year of adoption as standard or, in the case of revision, the year of last revision. The serial designations of Standards, which have for any reason been discontinued, are permanently dropped. In ordering Standards, the complete serial designations should be stated, thus, A 1 – 27, A 2 – 27, etc.

The standards are grouped according to the materials to which they apply. Duplicate listings appear in italics.

A. FERROUS METALS

STEEL

Standard Specifications

Steel Rails and Accessories.

A 1 – 27. For Open-Hearth Carbon-Steel Rails.
A 2 – 27. For the Manufacture of Open-Hearth Steel Girder Rails of Plain, Grooved and Guard Types.
A 3 – 24. For Low-Carbon-Steel Splice Bars.
A 4 – 14. For Medium-Carbon-Steel Splice Bars.
A 5 – 14. For High-Carbon-Steel Splice Bars.
A 6 – 14. For Extra-High-Carbon-Steel Splice Bars.
A 49 – 21. For Quenched High-Carbon-Steel Splice Bars.
A 76 – 27. For Low-Carbon-Steel Track Bolts.
A 50 – 24. For Quenched Carbon-Steel Track Bolts.
A 51 – 21. For Quenched Alloy-Steel Track Bolts.
A 65 – 24. For Steel Track Spikes.
A 66 – 21. For Steel Screw Spikes.

Structural Steel.

A 7 – 24. For Structural Steel for Bridges.
A 8 – 24. For Structural Nickel Steel.
A 94 – 27. For Structural Silicon Steel.
A 9 – 24. For Structural Steel for Buildings.
A 10 – 24. For Structural Steel for Locomotives.
A 11 – 24. For Structural Steel for Cars.
A 12 – 21. For Structural Steel for Ships.
A 13 – 24. For Rivet Steel for Ships.

Boiler Steels.

A 30 – 24. For Boiler and Firebox Steel for Locomotives.
A 70 – 27. For Boiler and Firebox Steel for Stationary Service.
A 31 – 24. For Boiler Rivet Steel.

Steel for Welding.

A 78 – 27. For Steel Plates of Structural Quality for Forge Welding.
A 89 – 27. For Steel Plates of Flange Quality for Forge Welding.

Concrete Reinforcement Steel.

A 15 – 14. For Billet-Steel Concrete Reinforcement Bars.
A 16 – 14. For Rail-Steel Concrete Reinforcement Bars.
A 82 – 27. For Cold-Drawn Steel Wire for Concrete Reinforcement.

Bar Steels.

A 107 – 27. For Commercial Quality Hot-Rolled Bar Steels.
A 108 – 27. For Commercial Cold-Finished Bar Steels and Cold-Finished Shafting.

Spring Steel and Springs.

A 14 – 27. For Carbon-Steel Bars for Railway Springs.
A 68 – 27. For Carbon-Steel Bars for Railway Springs with Special Silicon Requirements.
A 58 – 27. For Carbon-Steel Bars for Vehicle and General Purpose Springs.
A 59 – 27. For Silico-Manganese-Steel Bars for Railway Springs.
A 60 – 27. For Chrome-Vanadium-Steel Bars for Railway Springs.
A 61 – 16. For Helical Steel Springs for Railways.
A 62 – 16. For Elliptical Steel Springs for Railways.

Steel Blooms, Forgings and Axles.

*A 17 – 21. For Carbon-Steel and Alloy-Steel Blooms, Billets and Slabs for Forgings.
A 18 – 27. For Carbon-Steel and Alloy-Steel Forgings.
A 19 – 27. For Quenched-and-Tempered Carbon-Steel Axles, Shafts, and Other Forgings for Locomotives and Cars.
A 63 – 27. For Quenched-and-Tempered Alloy-Steel Axles, Shafts, and Other Forgings for Locomotives and Cars.
A 20 – 27. For Carbon-Steel Forgings for Locomotives.
A 21 – 27. For Carbon-Steel Car and Tender Axles.
A 22 – 21. For Cold-Rolled Steel Axles.

* Approved as "Tentative American Standard" by the American Engineering Standards Committee.

Steel Chain.

A 56 – 24. For Iron and Steel Chain.

Steel Wheels and Tires.

A 25 – 24. For Wrought Solid Carbon-Steel Wheels for Electric Railway Service.
A 57 – 24. For Wrought Solid Carbon-Steel Wheels for Steam Railway Service.
A 26 – 16. For Steel Tires.

Steel Castings.

A 27 – 24. For Steel Castings.
A 87 – 27. For Carbon-Steel Castings for Railroads.

Steel Tubes and Pipe.

A 83 – 27. For Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes.
A 53 – 27. For Welded and Seamless Steel Pipe.

Tool Steels.

A 71 – 26. For Carbon Tool Steel.
A 92 – 26. For High-Speed Tool Steel.

Steel for High-Temperature Service.

A 96 – 27. For Alloy-Steel Bolting Material for High-Temperature Service.

Zinc-Coated Sheets.

A 93 – 27. For Zinc-Coated (Galvanized) Sheets.

STANDARD METHODS

A 33 – 24. Of Chemical Analysis of Plain Carbon Steel.
A 55 – 24. Of Chemical Analysis of Alloy Steels.
A 90 – 24. Of Determining Weight of Coating on Zinc-Coated Articles.
A 91 – 24. Of Determining Weight of Coating on Tin, Terne, and Lead-Coated Sheets.
A 34 – 27. Of Test for Magnetic Properties of Iron and Steel.
E 3 – 24. Of Metallographic Testing of Iron and Steel.
E 10 – 27. Of Brinell Hardness Testing of Metallic Materials.

RECOMMENDED PRACTICES

A 35 – 24. For Annealing of Miscellaneous Rolled and Forged Carbon-Steel Objects.
A 36 – 24. For Heat Treatment of Carbon-Steel Castings.
A 37 – 27. For Carburizing and Heat Treatment of Carburized Objects.

WROUGHT IRON

STANDARD SPECIFICATIONS

A 72 – 27. For Welded Wrought-Iron Pipe.
A 83 – 27. *For Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes.*
A 84 – 27. For Staybolt, Engine-Bolt and Extra-Refined Wrought-Iron Bars.
A 86 – 27. For Hollow Rolled Staybolt Iron.
*A 41 – 18. For Refined Wrought-Iron Bars.
A 85 – 27. For Common Iron Bars.
*A 42 – 18. For Wrought-Iron Plates.
A 73 – 24. For Wrought-Iron Rolled or Forged Blooms and Forgings for Locomotives and Cars.
A 56 – 24. For Iron and Steel Chain.

STANDARD DEFINITIONS

A 81 – 27. Of Terms Relating to Wrought-Iron Specifications.

PIG IRON, CAST IRON, AND FINISHED CASTINGS

STANDARD SPECIFICATIONS

A 43 – 24. For Foundry Pig Iron.
A 44 – 04. For Cast-Iron Pipe and Special Castings.
A 74 – 18. For Cast-Iron Soil Pipe and Fittings.
A 45 – 14. For Cast-Iron Locomotive Cylinders.
A 46 – 24. For Chilled Cast-Iron Wheels.
A 47 – 27. For Malleable Castings.
A 48 – 18. For Gray-Iron Castings.
A 88 – 24. For High-Test Gray-Iron Castings.

STANDARD METHODS

A 64 – 27. Of Sampling and Chemical Analysis of Pig and Cast Iron.

FERRO-ALLOYS

STANDARD SPECIFICATIONS

A 97 – 27. For Tungsten Powder.
A 98 – 27. For Spiegeleisen.
A 99 – 27. For Ferro-Manganese.
A 100 – 27. For Ferro-Silicon.
A 101 – 27. For Ferro-Chromium.
A 102 – 27. For Ferro-Vanadium.

STANDARD METHODS

A 103 – 27. Of Sampling Ferro-Alloys.
A 104 – 27. Of Chemical Analysis of Ferro-Alloys.

* Approved as "Tentative American Standard" by the American Engineering Standards Committee.

B. NON-FERROUS METALS

STANDARD SPECIFICATIONS

Ingot Copper.

B 4 – 27. For Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars.
B 5 – 27. For Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars.

Spelter.

B 6 – 18. For Spelter.

Lead.

B 29 – 23. For Pig Lead.

Nickel.

B 39 – 22. For Nickel.

Aluminum.

B 37 – 26. For Aluminum for Use in the Manufacture of Iron and Steel.

Deoxidizers.

B 51 – 27. For Phosphor Tin.
B 52 – 27. For Phosphor Copper.
B 53 – 27. For Silicon Copper.

Wire and Cable.

B 49 – 26. For Hot-Rolled Copper Rods for Wire Drawing.
B 1 – 27. For Hard-Drawn Copper Wire.
B 2 – 27. Medium Hard-Drawn Copper Wire.
B 3 – 27. For Soft or Annealed Copper Wire.
B 33 – 21. For Tinned Soft or Annealed Copper Wire for Rubber Insulation.
B 48 – 27. For Soft Rectangular Copper Wire.
B 8 – 27. For Bare Concentric-Lay Copper Cable: Hard, Medium-Hard, or Soft.
B 47 – 27. For Round and Grooved Hard-Drawn Copper Trolley Wire.

Bronze.

B 10 – 18. For the Alloy: Copper, 88 per cent; Tin, 10 per cent; Zinc, 2 per cent.
*B 30 – 22. For Brass Ingot Metal, Graded and Ungraded, for Sand Castings.
B 31 – 21. For Bronze Bearing Metal in Ingot Form.
B 22 – 21. For Bronze Bearing Metals for Turntables and Movable Railroad Bridges.
B 7 – 27. For Manganese-Bronze Ingots for Sand Castings.
B 54 – 27. For Manganese-Bronze Sand Castings.

* Approved as "Tentative American Standard" by the American Engineering Standards Committee.

White Metal. *

*B 32 – 21. For Solder Metal.
B 23 – 26. For White Metal Bearing Alloys (known commercially as "Babbitt Metal").

Copper and Brass Plates, Tubes, Rods, etc.

B 11 – 18. For Copper Plates for Locomotive Fireboxes.
B 12 – 21. For Copper Bars for Locomotive Staybolts.
B 13 – 18. For Seamless Copper Boiler Tubes.
B 14 – 18. For Seamless Brass Boiler Tubes.
B 44 – 24. For Seamless Admiralty Condenser Tubes and Ferrule Stock.
B 55 – 25. For Seamless 70–30 Brass Condenser Tubes and Ferrule Stock.
B 56 – 25. For Seamless Muntz Metal Condenser Tubes and Ferrule Stock.
B 57 – 27. For Muntz Metal Condenser Tube Plates.
B 42 – 24. For Copper Pipe, Standard Sizes.
B 43 – 24. For Brass Pipe, Standard Sizes.
*B 15 – 18. For Brass Forging Rod.
*B 16 – 18. For Free-Cutting Brass Rod for Use in Screw Machines.
B 19 – 19. For Cartridge Brass.
B 20 – 19. For Cartridge Brass Disks.
B 21 – 27. For Naval Brass Rods for Structural Purposes.
B 36 – 27. For Sheet High Brass.

STANDARD METHODS

*B 34 – 20. Of Battery Assay of Copper.
B 35 – 24. Of Chemical Analysis of Pig Lead.
B 38 – 21. Of Chemical Analysis of Spelter.
B 41 – 23. Of Chemical Analysis of Nickel.
*B 18 – 21. Of Chemical Analysis of Alloys of Lead, Tin, Antimony and Copper.
*B 27 – 19. Of Chemical Analysis of Manganese Bronze.
*B 28 – 19. Of Chemical Analysis of Gun Metal.
B 45 – 27. Of Chemical Analysis of Brass Ingots and Sand Castings.
B 46 – 27. Of Chemical Analysis of Bronze Bearing Metal.
A 90 – 24. Of Determining Weight of Coating on Zinc-Coated Articles.
A 91 – 24. Of Determining Weight of Coating on Tin, Terne, and Lead-Coated Sheets.
E 5 – 27. Of Metallographic Testing of Non-Ferrous Metals and Alloys.
E 10 – 27. Of Brinell Hardness Testing of Metallic Materials.

C. CEMENT, LIME, GYPSUM, CONCRETE, AND CLAY PRODUCTS

CEMENT

STANDARD SPECIFICATIONS

C 9 – 26. Specifications and Tests for Portland Cement.
C 10 – 09. For Natural Cement.

* Approved as "Tentative American Standard" by the American Engineering Standards Committee.

LIME

STANDARD SPECIFICATIONS

C 5 – 26. For Quicklime for Structural Purposes.
C 6 – 24. For Hydrated Lime for Structural Purposes.
C 45 – 25. For Quicklime and Hydrated Lime for Use in the Cooking of Rags for the Manufacture of Paper.
C 46 – 27. For Quicklime for Use in the Manufacture of Sulfite Pulp.
C 47 – 27. For Hydrated Lime for the Manufacture of Varnish.
C 48 – 24. For Quicklime and Hydrated Lime for Use in the Textile Industry.
C 49 – 24. For Quicklime and Hydrated Lime for the Manufacture of Silica Brick.
C 53 – 27. For Quicklime for Use in Water Treatment.
C 54 – 27. For Hydrated Lime for Use in Water Treatment.

STANDARD METHODS

C 50 – 27. Of Sampling, Inspection, Packing and Marking of Quicklime and Lime Products.

GYPSUM

STANDARD SPECIFICATIONS

C 22 – 25. For Gypsum.
C 23 – 22. For Calcined Gypsum.
C 28 – 27. For Gypsum Plasters.
C 35 – 25. For Gypsum Plastering Sand.
C 36 – 25. For Gypsum Wall Board.
C 37 – 25. For Gypsum Plaster Board.
C 52 – 27. For Gypsum Partition Tile or Block.

STANDARD METHODS

C 26 – 27. Of Testing Gypsum and Gypsum Products.

STANDARD DEFINITIONS

C 11 – 26. Of Terms Relating to the Gypsum Industry.

CONCRETE AND CONCRETE AGGREGATES

STANDARD SPECIFICATIONS

D 57 – 20. For Materials for Cement Grout Filler for Brick and Stone Block Pavements.
D 58 – 24. For Materials for Cement Mortar Bed for Brick, Stone Block, Wood Block, Asphalt Block and Other Block Pavements.

STANDARD METHODS

C 31 – 27. Of Making and Storing Specimens of Concrete in the Field.
C 39 – 27. Of Making Compression Tests of Concrete.
C 42 – 27. Of Securing Specimens of Hardened Concrete from the Structure.
C 29 – 27. Of Test for Unit Weight of Aggregate for Concrete.

*C 30 – 22. Of Test for Determination of Voids in Fine Aggregate for Concrete.
C 40 – 27. Of Test for Organic Impurities in Sands for Concrete.
C 41 – 24. Of Test for Sieve Analysis of Aggregates for Concrete.
*D 30 – 18. *Of Test for Apparent Specific Gravity of Coarse Aggregates.*
*D 55 – 25. *Of Test for Apparent Specific Gravity of Sand, Stone and Slag Screenings, and Other Fine Non-Bituminous Highway Materials.*

BRICKS AND REFRACTORIES

STANDARD SPECIFICATIONS

C 7 – 15. For Paving Brick.
C 32 – 24. For Clay Sewer Brick.

STANDARD METHODS

C 16 – 20. Of Test for Refractory Materials Under Load at High Temperatures.
C 20 – 20. Of Test for Porosity and Permanent Volume Changes in Refractory Materials.
C 24 – 20. Of Test for Softening Point of Fire-Clay Brick.
C 18 – 21. Of Ultimate Chemical Analysis of Refractory Materials, Including Chrome Ores and Chrome Brick.

STANDARD DEFINITIONS

C 27 – 20. For Clay Refractories.

PIPE AND DRAIN TILE

STANDARD SPECIFICATIONS

C 13 – 24. For Clay Sewer Pipe.
C 14 – 24. For Cement-Concrete Sewer Pipe.
†C 4 – 24. For Drain Tile.

STANDARD DEFINITIONS

C 8 – 24. Of Terms Relating to Sewer Pipe.

RECOMMENDED PRACTICE

C 12 – 19. For Laying Sewer Pipe.

HOLLOW TILE

STANDARD SPECIFICATIONS

C 34 – 27. Specifications and Tests for Hollow Burned-Clay Load-Bearing Wall Tile.
C 57 – 27. Specifications and Tests for Hollow Burned-Clay Floor Tile.

STANDARD DEFINITIONS

C 43 – 24. Of Terms Relating to Hollow Tile.

* Approved as "Tentative American Standard" by the American Engineering Standards Committee.
† Approved as "American Standard" by the American Engineering Standards Committee.

MISCELLANEOUS SUBJECTS

STANDARD SPECIFICATIONS

E 11 – 26. *For Sieves for Testing Purposes.*

STANDARD DEFINITIONS

E 12 – 27. *Of Terms Relating to Specific Gravity.*

D. MISCELLANEOUS MATERIALS

PRESERVATIVE COATINGS

STANDARD SPECIFICATIONS

D 234 – 27. For Raw Linseed Oil.
D 125 – 23. For Perilla Oil, Raw or Refined.
D 13 – 26. For Gum Spirits of Turpentine and Steam-Distilled Wood Turpentine.
D 236 – 27. For Destructively Distilled Wood Turpentine.
D 207 – 26. For Dry Bleached Shellac.
D 237 – 27. For Orange Shellac.
D 79 – 24. For Zinc Oxide.
D 80 – 24. For Leaded Zinc Oxide.
D 81 – 24. For Basic Carbonate White Lead.
D 82 – 24. For Basic Sulfate White Lead.
D 83 – 24. For Red Lead.
D 84 – 27. For Mineral Iron Oxide.
D 85 – 27. For Ocher.
D 208 – 26. For Lithopone.
D 209 – 26. For Lampblack.
D 210 – 26. For Bone Black.
D 211 – 27. For Chrome Yellow.
D 212 – 27. For Pure Chrome Green.
D 213 – 27. For Reduced Chrome Green.

STANDARD METHODS

D 233 – 26. Of Sampling and Testing Turpentine.
D 153 – 27. Of Test for Specific Gravity of Pigments.
D 185 – 26. Of Test for Coarse Particles in Paint Pigments.
D 154 – 25. Of Testing Oleo-Resinous Varnishes.
D 29 – 25. Of Testing Shellac.
*D 56 – 21. Of Test for Flash Point of Volatile Flammable Liquids.
*D 34 – 27. Of Routine Analysis of White Pigments.
*D 49 – 27. Of Routine Analysis of Dry Red Lead.
*D 50 – 27. Of Routine Analysis of Yellow, Orange, Red and Brown Pigments Containing Iron and Manganese.
D 126 – 27. Of Routine Analysis of Yellow and Orange Pigments Containing Chromium Compounds, Blue Pigments and Chrome Green.
D 186 – 27. Of Routine Analysis of Titanium Pigments.

STANDARD DEFINITIONS

D 16 – 24. Of Terms Relating to Paint Specifications.

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* Approved as "Tentative American Standard" by the American Engineering Standards Committee.

PETROLEUM PRODUCTS AND LUBRICANTS

See also **Road Materials** and **Waterproofing and Roofing Materials**

STANDARD METHODS

D 206 – 25. Abridged Volume Correction Table for Petroleum Oils.
D 128 – 27. Of Analysis of Grease.
D 187 – 27. Of Test for Burning Quality of Kerosine Oils.
D 219 – 27. Of Test for Burning Quality of Long-Time Burning Oil for Railway Use.
D 239 – 27. Of Test for Burning Quality of Mineral Seal Oil.
D 86 – 27. Of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products.
D 216 – 27. Of Test for Distillation of Natural Gas Gasoline.
D 117 – 27. Of Testing Electrical Insulating Oils.
D 56 – 21. Of Test for Flash Point of Volatile Flammable Liquids.
D 93 – 22. Of Test for Flash Point by Means of the Pensky-Martens Closed Tester.
D 92 – 24. Of Test for Flash and Fire Points by Means of Open Cup.
D 87 – 22. Of Test for Melting Point of Paraffin Wax.
D 157 – 27. Of Test for Steam Emulsion of Lubricating Oils.
D 129 – 27. Of Test for Sulfur in Petroleum Oils Heavier than Illuminating Oil.
D 240 – 27. Of Test for Thermal Value of Fuel Oil.
D 88 – 26. Of Test for Viscosity of Petroleum Products and Lubricants.
D 95 – 27. Of Test for Water in Petroleum Products and Other Bituminous Materials.
D 96 – 24. Of Test for Water and Sediment in Petroleum Products by Means of Centrifuge.

ROAD MATERIALS

STANDARD SPECIFICATIONS

*D 57 – 20. For Materials for Cement Grout Filler for Brick and Stone Block Pavements.
D 58 – 24. For Materials for Cement Mortar Bed for Brick, Stone Block, Wood Block, Asphalt Block and Other Block Pavements.
C 7 – 15. For Paving Brick.
D 59 – 26. For Block for Granite Block Pavements.
D 131 – 23. For Block for Recut Granite Block Pavements.
D 132 – 23. For Block for Durax Granite Pavements.
D 52 – 20. For Wooden Paving Blocks for Exposed Pavements.
D 65 – 23. For Broken Slag for Waterbound Base and Wearing Course.
D 66 – 23. For Shovel-Run or Crusher-Run Broken Slag for Waterbound Base.
D 195 – 27. For Broken Slag for Bituminous Macadam Base.
D 196 – 27. For Broken Slag for Bituminous Concrete Base.
D 159 – 27. For Broken Slag for Bituminous Macadam Wearing Course.

* Approved as "Tentative American Standard" by the American Engineering Standards Committee.

D 160 – 27. For Broken Slag for Bituminous Concrete (Coarse-Graded Aggregate Type).
D 161 – 27. For Broken Slag for Bituminous Concrete (Fine-Graded Aggregate Type).
E 11 – 26. For Sieves for Testing Purposes.

STANDARD METHODS

D 2 – 26. Of Test for Abrasion of Rock.
*D 3 – 18. Of Test for Toughness of Rock.
*D 30 – 18. Of Test for Apparent Specific Gravity of Coarse Aggregates.
D 55 – 25. Of Test for Apparent Specific Gravity of Sand, Stone and Slag Screenings, and Other Fine Non-Bituminous Highway Materials.
D 72 – 21. Of Test for Quantity of Clay and Silt in Gravel for Highway Construction.
*D 75 – 22. Of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods.
D 7 – 27. Of Mechanical Analysis of Sand or Other Fine Highway Material, Except Fine Aggregates Used in Cement Concrete.
D 18 – 16. Of Mechanical Analysis of Broken Stone or Broken Slag, Except Aggregates Used in Cement Concrete.
D 19 – 16. Of Mechanical Analysis of Mixtures of Sand or Other Fine Material with Broken Stone or Broken Slag, Except Aggregates Used in Cement Concrete.
D 35 – 18. Form of Specifications for Certain Commercial Grades of Broken Stone.
D 4 – 27. Of Test for the Determination of Bitumen.
D 165 – 27. Of Test for the Determination of Proportion of Bitumen Soluble in Carbon Tetrachloride.
D 6 – 27. Of Test for Loss on Heating of Oil and Asphaltic Compounds.
D 5 – 25. Of Test for Penetration of Bituminous Materials.
D 36 – 26. Of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method).
D 61 – 24. Of Test for Softening Point of Tar Products (Cube-in-Water Method).
D 139 – 27. Of Float Test for Bituminous Materials.
D 70 – 27. Of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements and Soft Tar Pitches.
D 71 – 27. Of Test for Specific Gravity of Asphalts and Tar Pitches Sufficiently Solid to be Handled in Fragments.
D 140 – 25. Of Sampling Bituminous Materials.
D 95 – 27. Of Test for Water in Petroleum Products and Other Bituminous Materials.

STANDARD DEFINITIONS

D 8 – 18. Of Terms Relating to Materials for Roads and Pavements.
E 12 – 27. Of Terms Relating to Specific Gravity.

* Approved as "Tentative American Standard" by the American Engineering Standards Committee.

COAL AND COKE

STANDARD SPECIFICATIONS

D 17 – 16. For Foundry Coke.
D 166 – 24. For Gas and Coking Coals.

STANDARD METHODS

*D 21 – 16. Of Sampling Coal.
D 271 – 27. Of Laboratory Sampling and Analysis of Coal and Coke.
D 141 – 23. Of Shatter Test for Coke.
D 167 – 24. Of Test for Volume of Cell Space of Lump Coke.

STANDARD DEFINITIONS

D 121 – 27. Of Terms Relating to Coal and Coke.

TIMBER AND TIMBER PRESERVATIVES

STANDARD SPECIFICATIONS

D 245 – 27. For Structural Wood Joist, Planks, Beams, Stringers and Posts
D 25 – 20. For Southern Yellow-Pine Piles and Poles to be Creosoted.
D 52 – 20. For Wooden Paving Blocks for Exposed Pavements.

STANDARD METHODS

D 143 – 27. Of Testing Small Clear Specimens of Timber.
D 198 – 27. For Conducting Static Tests of Timber in Structural Sizes.
D 38 – 27. Of Sampling and Analysis of Creosote Oil.
D 168 – 27. Of Test for Coke Residue of Creosote Oil.
D 199 – 27. Of Chemical Analysis of Zinc Chloride.

STANDARD DEFINITIONS

D 9 – 15. Of Terms Relating to Structural Timber.

WATERPROOFING AND ROOFING MATERIALS

STANDARD SPECIFICATIONS

D 40 – 25. For Asphalt for Use in Damp-proofing and Waterproofing Below Ground Level.
D 144 – 25. For Asphalt for Use in Damp-proofing and Waterproofing Above Ground Level.
D 41 – 26. For Primer for Use with Asphalt in Damp-proofing and Waterproofing Below and Above Ground Level.
D 42 – 25. For High-Carbon Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level.
D 145 – 25. For High-Carbon Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Above Ground Level.
D 200 – 25. For High-Bitumen Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level.

* Approved as "Tentative American Standard" by the American Engineering Standards Committtee.

D 201 – 25. For High-Bitumen Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Above Ground Level.
D 251 – 27. For High-Carbon Coal-Tar Pitch for Use in Constructing Built-Up Roofs Surfaced with Slag or Gravel.
D 252 – 27. For High-Bitumen Coal-Tar Pitch for Use in Constructing Built-Up Roofs Surfaced with Slag or Gravel.
D 43 – 25. For Creosote Oil for Priming Coat with Coal-Tar Pitch in Damp-proofing and Waterproofing Below and Above Ground Level.
D 169 – 25. For Asphalt Mastic for Use in Waterproofing.
D 170 – 25. For Bituminous Grout for Use in Waterproofing Above Ground Level.
D 171 – 25. For Bituminous Grout for Use in Waterproofing Below Ground Level.
D 173 – 27. For Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing.
D 174 – 25. For Burlap Saturated with Bituminous Substances for Use in Waterproofing.
D 224 – 27. For Asphalt Roll-Roofing Surfaced with Powdered Talc.
D 225 – 27. For Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles.
D 248 – 27. For Asphalt Roll-Roofing Surfaced with Granular Talc.
D 249 – 27. For Heavy Weight Slate-Surfaced Asphalt Roll-Roofing and Heavy Weight Slate-Surfaced Asphalt Shingles.
D 226 – 27. For Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs.
D 227 – 27. For Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs.
D 250 – 27. For Asphalt-Saturated Asbestos Felt for Use in Constructing Built-Up Roofs.

STANDARD METHODS

D 147 – 27. Of Testing Bituminous Mastics, Grouts and Like Mixtures.
D 146 – 27. Of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing.
D 228 – 27. Of Testing Asphalt Roll-Roofing Surfaced with Fine or Granular Talc, Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles.

SLATE

STANDARD DEFINITION

D 247 – 27. Of the Term Slate.

INSULATING MATERIALS

STANDARD SPECIFICATIONS

D 120 – 23. For Rubber Gloves for Electrical Workers on Apparatus or Circuits Not Exceeding 3000 Volts to Ground.
D 178 – 24. For Rubber Matting for Use Around Electrical Apparatus or Circuits Not Exceeding 3000 Volts to Ground.
D 69 – 24. For Adhesive Tape for General Use for Electrical Purposes.

D 259 – 27. For Tolerances and Test Methods for Electrical Silk and Cotton Tapes.
D 203 – 25. For Tolerances and Test Methods for Electrical Cotton Yarns.

STANDARD METHODS

D 48 – 27. Of Testing Molded Insulating Materials.
D 117 – 27. Of Testing Electrical Insulating Oils.

RUBBER PRODUCTS

STANDARD SPECIFICATIONS

D 14 – 23. For Cotton Rubber-Lined Fire Hose for Private Fire Department Use.
D 26 – 23. For Cotton Rubber-Lined Fire Hose for Public Fire Department Use.
D 46 – 24. For Wrapped Air Hose for Use with Pneumatic Tools.
D 60 – 24. For Braided Air Hose for Use with Pneumatic Tools.
D 177 – 24. For Wrapped Cold Water Hose.
D 53 – 24. For Rubber Belting for Power Transmission.
D 120 – 23. For Rubber Gloves for Electrical Workers on Apparatus or Circuits Not Exceeding 3000 Volts to Ground.
D 178 – 24. For Rubber Matting for Use Around Electrical Apparatus or Circuits Not Exceeding 3000 Volts to Ground.
D 151 – 23. For Rubber Pump Valves.
D 69 – 24. For Adhesive Tape for General Use for Electrical Purposes.

STANDARD METHODS

D 15 – 24. Of Testing Rubber Products.

TEXTILE MATERIALS

STANDARD SPECIFICATIONS

D 76 – 27. For Textile Testing Machines.
D 180 – 27. For Tolerances and Test Methods for Cotton Yarns, Single and Plied.
D 203 – 25. For Tolerances and Test Methods for Electrical Cotton Yarns.
D 204 – 27. For Tolerances and Test Methods for Cotton Sewing Threads.
D 230 – 27. For Tolerances for Numbered Cotton Duck.
D 122 – 27. For Tolerances and Test Methods for Tire Fabrics Other Than Cord Fabrics.
D 179 – 27. For Tolerances and Test Methods for Cord Tire Fabrics.
D 181 – 25. For Tolerances for Hose Ducks and Belt Ducks.
D 259 – 27. For Tolerances and Test Methods for Electrical Silk and Cotton Tapes.
D 205 – 27. Specifications and Tests for Osnaburg Cement Sacks.

STANDARD METHODS

D 39 – 27. Of Testing Woven Textile Fabrics.

STANDARD DEFINITIONS

D 123 – 27. Of Terms Relating to Textile Materials.

THERMOMETERS

STANDARD SPECIFICATIONS

D 182 – 25. For A.S.T.M. Partial-Immersion Thermometer for General Use, −20 to +150° C., 0 to +300° F.
D 183 – 25. For A.S.T.M. Partial-Immersion Thermometer for General Use, −5 to +300° C., +20 to +580° F.
D 184 – 25. For A.S.T.M. Partial-Immersion Thermometer for General Use, −5 to +400° C., +20 to +760° F.

E. MISCELLANEOUS SUBJECTS

STANDARD SPECIFICATIONS

E 11 – 26. For Sieves for Testing Purposes.

STANDARD METHODS

E 4 – 27. Of Verification of Testing Machines.
E 3 – 24. Of Metallographic Testing of Iron and Steel.
E 5 – 27. Of Metallographic Testing of Non-Ferrous Metals and Alloys.
E 10 – 27. Of Brinell Hardness Testing of Metallic Materials.

STANDARD RULES

E 2 – 27. Governing the Preparation of Micrographs of Metals and Alloys, Including Recommended Practice for Photography as Applied to Metallography.

STANDARD DEFINITIONS

E 7 – 27. Of Terms Relating to Metallography.
E 12 – 27. Of Terms Relating to Specific Gravity.

LIST OF A.S.T.M. TENTATIVE STANDARDS

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A. FERROUS METALS

TENTATIVE SPECIFICATIONS

A 65 – 26 T.	For Soft Steel Track Spikes.[1]
A 67 – 26 T.	For Steel Tie Plates.
A 77 – 20 T.	For Electric Cast-Steel Anchor Chain.
A 113 – 27 T.	For Structural Steel for Locomotives and Cars.
A 114 – 27 T.	For Marine Boiler Steel Plates.
A 95 – 26 T.	For Carbon-Steel Castings for Valves, Flanges, and Fittings for High-Temperature Service.
A 105 – 27 T.	For Forged or Rolled Steel Pipe Flanges for High-Temperature Service.

[1] To replace, when adopted, the present Standard Specifications for Steel Track Spikes (Serial Designation: A 65 – 24).

A 106 – 27 T. For Lap-Welded and Seamless Steel Pipe for High-Temperature Service.
A 109 – 27 T. For Cold-Rolled Strip Steel.
A 115 – 27 T. For Alloy Tool Steel.
A 111 – 27 T. For Zinc-Coated (Galvanized) Iron or Steel Telephone and Telegraph Line Wire.
A 112 – 27 T. For Zinc-Coated (Galvanized) Iron or Steel Tie Wires.
A 116 – 27 T. For Zinc-Coated (Galvanized) Wire Fencing.
A 117 – 27 T. For Zinc-Coated Chain-Link Fence Fabric Galvanized After Weaving.
A 118 – 27 T. For Zinc-Coated Chain-Link Fence Fabric Galvanized Before Weaving.

TENTATIVE METHODS

A 110 – 27 T. Of Testing Zinc-Coated (Galvanized) Iron and Steel Wire and Wire Products.
A 34 – 27 T. Of Test for Magnetic Properties of Iron and Steel at Low Inductions for Audio and Power Frequencies.[1]
E 8 – 27 T. Of Tension Testing of Metallic Materials.
E 9 – 27 T. Of Compression Testing of Metallic Materials.

TENTATIVE RECOMMENDED PRACTICE

E 15 – 26 T. For Radiographic Testing of Metal Castings.

TENTATIVE DEFINITIONS

A 119 – 27 T. Of Terms Relating to Heat Treatment Operations (Especially as Related to Ferrous Alloys).

B. NON-FERROUS METALS

TENTATIVE SPECIFICATIONS

B 64 – 27 T. For Brazing Solder.
B 58 – 26 T. For Aluminum-Base Sand-Casting Alloys in Ingot Form.
B 26 – 26 T. For Aluminum Base Alloy Sand Castings.
D 27 – 21 T. For Insulated Wire and Cable: 30-per-cent Hevea Rubber.
B 9 – 27 T. For Bronze Trolley Wire.
B 59 – 26 T. For Aluminum Bronze Castings.
B 60 – 26 T. For Sand Castings of the Alloy: Copper, 88 per cent; Tin, 8 per cent; Zinc, 4 per cent.
B 61 – 26 T. For Steam or Valve Bronze Sand Castings.
B 62 – 26 T. For Composition Brass or Ounce Metal Sand Castings.
B 65 – 27 T. For Yellow Brass Sand Castings for General Purposes.
B 66 – 27 T. For Bronze Castings in the Rough for Locomotive Wearing Parts.
B 67 – 27 T. For Car and Tender Journal Bearings, Lined.
B 24 – 26 T. For Aluminum Ingots for Remelting.

[1] To be added, when adopted, to the present Standard Methods of Test for Magnetic Properties of Iron and Steel (Serial Designation: A 34 – 27).

B 25 – 26 T. For Aluminum Sheet.
B 68 – 27 T. For Copper Tubing for Refrigerators.
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C 25 – 27 T. Of Chemical Analysis of Limestone, Quicklime and Hydrated Lime.

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C 51 – 24 T. Of Terms Relating to Lime.

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C 59 – 26 T. For Gypsum Molding Plaster.
C 60 – 26 T. For Gypsum Pottery Plaster.

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[1] To be added, when adopted, to the present Standard Specifications and Tests for Portland Cement (Serial Designation: C 9 – 26).

[2] To be added, when adopted, to the present Standard Definitions of Terms Relating to the Gypsum Industry (Serial Designation: C 11 – 26).

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[1] To replace, when adopted, the present Standard Specifications for Paving Brick (Serial Designation: C 7 – 15).

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* Approved as "Tentative American Standard" by the American Engineering Standards Committee.

[1] To be added, when adopted, to the present Standard Methods of Testing Oleo-Resinous Varnishes (Serial Designation: D 154 – 25).

[2] To be added, when adopted, to the present Standard Methods of Testing Shellac (Serial Designation: D 29 – 25).

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[1] To be added, when adopted, to the present Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (Serial Designation: D 271 – 27).

[2] To be added, when adopted, to the present Standard Definitions of Terms Relating to Coal and Coke (Serial Designation: D 121 – 27).

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[1] To be added, when adopted, to the present Standard Definitions of Terms Relating to Textile Materials (Serial Designation: D 123 – 27).

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RECOMMENDATIONS FOR MEMBERSHIP

AMERICAN SOCIETY FOR TESTING MATERIALS,
1315 Spruce Street,
Philadelphia, Pa.

Dear Sirs:

The undersigned recommends the following persons (or corporations, firms, etc.) for membership in the Society. It is suggested that you send them general information concerning the Society as well as application blanks for membership.

(In case of a firm, corporation, etc., indicate the name and title of the officer who should be addressed.)

Yours truly,

(Signed).....
Member, Am. Soc. Testing Mats.

Date.....

1927 B. S., II

American Society for Testing Materials

Application for Membership

THE UNDERSIGNED HEREBY APPLIES FOR { CORPORATE / INDIVIDUAL / JUNIOR } MEMBERSHIP IN THE AMERICAN SOCIETY FOR TESTING MATERIALS. IF THIS APPLICATION BE DULY APPROVED, HE AGREES TO BE GOVERNED BY THE CHARTER AND BY-LAWS OF THE SOCIETY AND TO FURTHER ITS OBJECTS AS LAID DOWN THEREIN.

NAME.....

(FIRM, ORGANIZATION OR INDIVIDUAL APPLICANT)

ADDRESS.....

.....

NATURE OF BUSINESS.....

IF CORPORATE MEMBERSHIP, NAME AND TITLE OF INDIVIDUAL WHO WILL EXERCISE MEMBERSHIP PRIVILEGES:

NAME..... TITLE.....

IF PERSONAL MEMBERSHIP, NAME OF ORGANIZATION WITH WHICH APPLICANT IS AFFILIATED AND OFFICIAL TITLE OR OCCUPATION:

NAME OF ORGANIZATION.....

ADDRESS OF ORGANIZATION.....

APPLICANT'S TITLE OR OCCUPATION.....

IF JUNIOR MEMBERSHIP GIVE DATE OF BIRTH.....

RECOMMENDED BY (TWO MEMBERS) .....

.....

[Application for membership should be mailed to AMERICAN SOCIETY FOR TESTING MATERIALS, 1315 Spruce Street, Philadelphia, Pa.]

1927 B. S., II

American Society for Testing Materials

Extract from Charter

1. The name of the proposed corporation is the "American Society for Testing Materials."

2. The corporation is formed for the promotion of knowledge of the materials of engineering, and the standardization of specifications and the methods of testing.

Extract from By-Laws

Article I. Members

Section 1. The Society shall consist of Members, Junior Members, Student Members, and Honorary Members.

Sec. 2. A Member shall be a person, or a corporation, a firm, a technical or scientific society, a teaching faculty, or a library, proposed by two members and approved by the Executive Committee.

Sec. 3. A Junior Member shall be a person less than twenty-seven years of age on the date of his admission, proposed by two members and approved by the Executive Committee. A Junior Member shall have the same rights and privileges as a Member, except that he shall not be eligible for office, and his status shall be changed from that of Junior Member to Member at the beginning of the fiscal year next succeeding the date on which he attains the age of twenty-seven years. * * *

Article V. Meetings

Section 1. The Society shall meet annually at a time and place fixed by the Executive Committee.

Sec. 2. Special meetings may be called whenever the Executive Committee shall deem it necessary, or upon the request in writing to the President of twenty-five (25) members.

Article VII. Dues

Section 1. The fiscal year shall commence on the first day of January. The annual dues, payable in advance, shall be as follows: For Members that are companies, firms, corporations, industrial associations or commercial testing laboratories, $30; for all other Members, $15; for Junior Members, $7.50; for Student Members, $3. Honorary Members shall not be subject to dues.[1]

Sec. 2. The entrance fees, payable on admission to the Society, shall be $10 for Members and $5 for Junior Members. The transfer fee, payable upon transfer from one grade of membership to another, shall be the difference between the corresponding entrance fees; provided that the payment of such transfer fee shall not be required of any Junior Member holding membership at the date of adoption of this section.

Sec. 5. Any person elected after six months of any fiscal year shall have expired, may pay only one-half of the amount of dues for that fiscal year; but in that case he shall not be entitled to a copy of the Proceedings for the current year.

[1] This Section of the By-laws was adopted September 1, 1927, to become effective January 1, 1928. Until the latter date the annual dues will continue to be: For Members (whether company or individual), $15; for Junior Members, $7.50; for Student Members, $3.

American Society for Testing Materials

Application for Student Membership

THE UNDERSIGNED HEREBY APPLIES FOR STUDENT MEMBERSHIP IN THE AMERICAN SOCIETY FOR TESTING MATERIALS AND ENCLOSES A REMITTANCE OF THREE DOLLARS IN PAYMENT OF THE CURRENT YEAR'S DUES. IF THIS APPLICATION BE DULY APPROVED, HE AGREES TO BE GOVERNED BY THE CHARTER AND BY-LAWS OF THE SOCIETY AND TO FURTHER ITS OBJECTS AS LAID DOWN THEREIN.

NAME OF APPLICANT .....

STUDENT IN (COURSE) .....

AT (NAME AND ADDRESS OF SCHOOL) .....

.....

YEAR (CHECK) JUNIOR, SENIOR OR GRADUATE

DATE OF BIRTH .....

ADDRESS FOR MAIL .....

.....

PROPOSED BY:

(SIGNED) .....

(TITLE) .....

MEMBER OF FACULTY

[Application for membership should be mailed to AMERICAN SOCIETY FOR TESTING MATERIALS, 1315 Spruce Street, Philadelphia, Pa.]

1927 B. S., II

American Society for Testing Materials

Extract from Charter

1. The name of the proposed corporation is the "American Society for Testing Materials."

2. The corporation is formed for the promotion of knowledge of the materials of engineering, and the standardization of specifications and the methods of testing.

Extract from By-Laws

Article I. Members

Section 1. The Society shall consist of Members, Junior Members, Student Members, and Honorary Members.

Sec. 2. A Member shall be a person, or a corporation, a firm, a technical or scientific society, a teaching faculty or a library, proposed by two members and approved by the Executive Committee.

Sec. 3. A Junior Member shall be a person less than twenty-seven years of age on the date of his admission, proposed by two members and approved by the Executive Committee. A Junior Member shall have the same rights and privileges as a Member, except that he shall not be eligible for office, and his status shall be changed from that of Junior Member to Member at the beginning of the fiscal year next succeeding the date on which he attains the age of twenty-seven years. * * *

Sec. 4. A Student Member shall be a Junior, Senior or Graduate Student in a technical school of recognized standing, or a student not over twenty-five years of age engaged in technical studies in an apprentice course or night school, proposed by a member of the faculty and elected by the Executive Committee.

A Student Member shall have such rights and privileges as may be determined by the Executive Committee and he shall not be eligible for office. His status shall be changed to that of Junior Member or Member upon severance of his connection with the technical school, apprentice course or night school.

Article V. Meetings

Section 1. The Society shall meet annually at a time and place fixed by the Executive Committee.

Sec. 2. Special meetings may be called whenever the Executive Committee shall deem it necessary, or upon the request in writing to the President of twenty-five (25) members.

Article VII. Dues

Section 1. The fiscal year shall commence on the first day of January. The annual dues, payable in advance, shall be as follows: For Members that are companies, firms, corporations, industrial associations or commercial testing laboratories, $30; for all other Members, $15; for Junior Members, $7.50; for Student Members, $3. Honorary Members shall not be subject to dues.[1]

Sec. 2. The entrance fees, payable on admission to the Society, shall be $10.00 for Members and $5.00 for Junior Members. The transfer fee, payable upon transfer from one grade of membership to another, shall be the difference between the corresponding entrance fees; provided that the payment of such transfer fee shall not be required of any Junior Member holding membership at the date of adoption of this section.

Resolution Adopted by Executive Committee

The Student Members shall receive the Year Book, Bulletins and other circular material, preprints upon the same basis as other members, and the triennial Book of Standards with the intervening annual supplements. Student Members shall have the privilege of other members of the Society of obtaining Society publications at special prices to members.

[1] This Section of the By-laws was adopted September 1, 1927, to become effective January 1, 1928. Until the latter date the annual dues will continue to be: For Members (whether company or individual), $15; for Junior Members, $7.50; for Student Members, $3.

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